

89B010A/3
OA dated 9/14/2006
Response dated 11/27/2006

REMARKS

Claims 1 to 122 are before the Examiner. Claims 1 to 26, 28 to 43, and 57 to 59 have been cancelled. Reconsideration of the Application is requested.

Claim 48 is amended so that it corresponds to the language in count 2 in Interference No. 102,953 (copy of count 2 enclosed, see page 1-3 of paper 21).

Claims 49 to 59 and 60 to 63, if amended, have been amended to change nomenclature to be consistent with the specific language in the claim 48 and/or to correct minor typographical errors.

New claims 64 to 122 have been added and are supported by the claims and specification as originally filed.

As the Examiner is aware, claims 2, 4-6, 25, 26, 35-41, 44 and 45 were the basis of Interference No. 102,953 and were held to correspond to Count 2. The Judgment in interference No. 102,953 states "Jo Ann M. Canich, the junior party, is entitled to a patent containing claims 2, 4-6, 25, 26, 35-41, 44 and 45 of their application corresponding to Count 2." (copy attached, see page 53 of paper no. 578)

Applicant requests the Examiner enter the following new claims 60 to 82 which are claims corresponding to claims 2, 4-6, 25, 26, 35-41, 44 and 45.

Specifically:

New Claim 64 corresponds to originally filed claim 2, except that it is now re-written in independent form.

New Claim 65 corresponds to originally filed claim 4 as amended in August of 1993, except that it is re-written in independent form.

New Claim 66 corresponds to originally filed claim 5, except that it is re-written in independent form.

New Claim 67 corresponds to originally filed claim 6, except that it is re-written in independent form.

New Claim 68 corresponds to claim 25.

New Claim 69 corresponds to claim 26.

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New Claim 70 correspond to Claim 35 as newly entered in August 1993.

New Claim 71 correspond to Claim 36 as newly entered in August 1993.

New Claim 72 correspond to Claim 37 as newly entered in August 1993.

New Claim 73 correspond to Claim 38 as newly entered in August 1993.

New Claim 74 correspond to Claim 39 as newly entered in August 1993.

New Claim 75 correspond to Claim 40 as newly entered in August 1993.

New Claim 76 correspond to Claim 41 as newly entered in August 1993.

New Claims 77 to 79 correspond to Claim 44, except that the dependency is no longer multiply dependent.

New Claims 80 to 82 correspond to Claim 45, except that the dependency is no longer multiply dependent.

The instant application is related to another application 07/676,690. Both have the same specification and are divisionals of the same parent case. As the Examiner is further aware, the second related application (USSN 07/676,690) was the basis for a related Interference No. 102,954. The Judgment in interference No. 102,954 states "Jo Ann M. Canich, the junior party, is entitled to claims 18-33 of their application, all corresponding to Count 2." (copy attached, see page 2 of paper 683 and page 3-4 of paper 681.) As the '690 application *was not* filed due to a restriction requirement and the instant application *was* filed due to a restriction requirement, Applicant herewith combines the claims, to avoid any possible double patenting issues.

Thus the Applicant requests the Examiner enter the following new claims 83 to 121 which are claims corresponding to or supported by claims 18-33 of USSN 07/676,690.

Specifically:

New Claims 83 to 87 correspond to Claims 18-33, but are written in dependent form.

New claims 88- 104 correspond to claims 18 to 33 that correspond to count 2 that Applicant was awarded in the Judgment in Interference 102, 954.

New claims 105- 120 are supported by original claims 18 to 33 and the specification as filed.

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New claim 121 corresponds to the specific language of count 2 in interference 102,954.

New claim 122 is supported by previous claim 27.

Applicant respectfully requests that the instant application be reconsidered in light of the above amendments and the following remarks.

Claim Objections

Claim 45 has been amended to correct "herein" to "wherein."

Claims 56 and 60 have been amended to correct "of" to "or."

Claim 62 has been amended to insert a period at the end.

Claim Rejections Under 35 USC § 112

Claims 27 and 44-63 are rejected under 35 USC § 112, second paragraph. Specifically Claim 27 is rejected for a muddled ligand attached to M'. Applicant has corrected the photocopying error. Claim 27 is also rejected for R' and M' not being defined and there being too many superfluous quotation marks. Applicant has corrected these typographical errors.

Claim 48 is rejected because y is 1 and thus there cannot be five substitutions on the Cp ring. Applicant has amended claim 48 to limit x to 0, 1, 2, 3, or 4.

Claim 53 is rejected as Ti was not within the scope of claim 48. Claim 48 now covers Ti.

Claim 68 is rejected because it depends from a cancelled claim. Applicant has changed the dependency to claim 48.

Applicant respectfully requests that the rejections be withdrawn.

Claim Rejections Under 35 USC § 102(e)

Claim 27 is rejected under 35 USC § 102(e) as anticipated by Example 1 of Tomotsu (US 5,276,117) which the Examiner suggest discloses Me₅CpTi(OMe)₃ with 1/2 molar equivalent of ethylene glycol. Claim 27 has been amended to define Q as being

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selected from the group consisting of halogen, hydride or C₁-C₂₀ hydrocarbyl. Since OMe is not a halogen, hydride or C1 to C20 hydrocarbyl, Tomatsu does not anticipate the claimed invention. Applicant respectfully requests that the rejection be withdrawn.

Claim Rejections Under 35 USC § 103(e)

Claims 27, 44 and 45 are rejected under 35 USC § 103(a) as anticipated by Examples 2 to 12 of Campbell (US 5,045,517) which the Examiner suggests discloses a variety of Cp complexes, including Cp-Ti-phenoxy dichloride. The Examiner acknowledges that L is missing, but suggests that one would be motivated to add L because Campbell suggest addition of alcohols at Column 2, line 31-39. Applicant respectfully disagrees, however to facilitate prosecution has amended the claim to delete "or an element with a coordination number of two from Group VI-A." Thus J cannot be phenoxy. Applicant respectfully requests that the rejection be withdrawn.

Claim Rejections Under 35 USC § 101 -Double Patenting

Claim 43 is provisionally rejected under 35 USC § 101 as claiming the same invention as claim 16 of USSN 07/963,833. Claim 43 has been cancelled.

Claim Rejections -Obviousness Type Double Patenting

Claims 48-63 are rejected as unpatentable under the judicially created doctrine of obviousness type double patenting over claims 1-3 and 5 of US 7,041,841.

Claims 48-63 are rejected as unpatentable under the judicially created doctrine of obviousness type double patenting over claims 1, 2 and 6-9 of US 5,621,126.

Claims 48-63 are rejected as unpatentable under the judicially created doctrine of obviousness type double patenting over claims 1-6 of RE 37,788.

Claims 48-63 are rejected as unpatentable under the judicially created doctrine of obviousness type double patenting over claims 1-20 of US 6,617,466.

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The cited patents and this application are commonly owned by ExxonMobil Chemical Patents Inc. (previously Exxon Chemical Patents Inc.).

Applicant respectfully disagrees with the above obviousness type double patenting rejections and respectfully submits that claims 48-63 are not obvious over the four cited patents and the rejections should be withdrawn.

In the instant application, the two-way test for obviousness type double patenting is to be applied. The two way test is applied only when the applicant could not have filed the claims in a single application *and* there is administrative delay. See *In re Berg*, 46 USPQ2d 1226 (Fed. Cir. 1998).

In particular, as explained in the U.S. Manual of Patent Examining Procedure (MPEP) §804 II B (b), if “the patent is the later filed application, the question of whether the timewise extension of the right to exclude granted by a patent is justified or unjustified must be addressed. A two-way test is to be applied only when the applicant could not have filed the claims in a single application *and* there is administrative delay. *In re Berg*, 46 USPQ2d 1226 (Fed. Cir. 1998) (“The two-way exception can only apply when the applicant could not avoid separate filings, and even then, only if the PTO controlled the rates of prosecution to cause the later filed species claims to issue before the claims for a genus in an earlier application . . .”)

MPEP § 804 further makes clear that when “making a two-way obviousness determination where appropriate, it is necessary to apply the *Graham* obviousness analysis twice, once with the application claims as the claims in issue, and once with the patent claims as the claims in issue. Where a two-way obviousness determination is required, an obvious-type double patenting rejection is appropriate only where each analysis compels a conclusion that the invention defined in the claims in issue is an obvious variation of the invention defined in a claim in the other application/patent. If either analysis does not compel a conclusion of obviousness, no double patenting rejection of the obvious-type is made...”

As further noted in MPEP § 804, a “delay in the processing of applications before the Office that would cause patents to issue in an order different from the order in which the applications were filed is a factor to be considered in determining whether a one-way or two-way obviousness determination is necessary to support a double patenting rejection” and “the resolution of legitimate differences of opinion that must be resolved... in an interference proceeding can significantly delay the issuance of a patent.”

Thus, as clearly noted in MPEP § 804, where “through no fault of the applicant, the claims in a later filed application issue first, an obvious-type double patenting rejection is improper, in the absence of a two-way obviousness determination, because the applicant does not have complete control over the rate of progress of a patent application through the Office. *In re Braat*, 937 F.2d 589, 19 USPQ2d 1289 (Fed. Cir. 1991). While acknowledging that allowance of the claims in the earlier filed application would result in the timewise extension of an invention claimed in the patent, the court was of the view that the extension was justified under the circumstances in this case, indicating that a double patenting rejection would be proper only if the claimed inventions were obvious over each other - a two-way obviousness determination.”

Applicant submits both elements triggering a two-way obviousness determination (applicant could not have filed the claims in a single application *and* there is administrative delay) are present in the instant application as compared to the four patents. The continued prosecution of the application beyond the issuance of the patents was not due to Applicant’s delay but rather due to the administrative delay in the USPTO, associated with interference proceedings of 1992-2004, (specifically Interferences 102, 953 and 102, 954).

Applicant further submits that the claims of the patents are patentably distinct from the claims of the present application and could not have been filed in the present application and thus the obviousness-type double patenting rejections should be withdrawn.

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Discussion of Obviousness-type Double Patenting rejections

Applicant was justified in defending the interference claims of this application and copending USSN 07/676,690 which resulted in the prosecution extending well beyond the issuance of the commonly owned patents named above. The patents were originally filed more than three months after the filing of the continuation-in-part base case of this application(533,245) and this application is a divisional of that CIP and was quickly filed after receipt of the restriction requirement, albeit later than the base applications for the patents US 5,264,405(incorrectly re-issued as US 6,265,338) and US 5,621,126 (reissued as RE37,788) but earlier than the base CIP case for US 7,041,841. Had the interference not delayed the instant application by more than a decade, Applicant has every reason to believe the case would have been timely prosecuted. In fact the instant application has been allowed several time in its history, and each time, the allowance was withdrawn by the USPTO. Thus Applicant submits that the administrative delay has been on the part of the USPTO and not the Applicant. A time line is listed below to illustrate the relationship of the application to the four patents above. For the Examiner's convenience a family tree is also attached.

Time Line

09/13/89 Original application 07/406,945;
06/04/90 CIP 07/533,245 of Original application 406,945--US 5,055,438;
09/19/90 CIP 07/581,841 of 533,245-- US 5,096,867
03/28/91 Divisional 07/676,690 of CIP 533,245 -- related to this application.
06/21/91 CIP 07/720,282 of CIP 533,245--now abandoned
07/11/91 Divisional 07/728,428 of CIP 533,245 -- **this application.**
08/28/91 Divisional 07/751,392 of CIP 581,869 -- US 5,227,440.
3/2/92 DIV 07/844,813 of 07/581,841 -- **US 6,617,466**
3/13/92 CIP 07/850,751 of 07/581,841--US 5,264,405 (now US 6,265,338).
11/9/92 DIV 07/973,261 of 07/720,282 -- **US 7041,841.**
10/15/93 CIP 08/138,169 of 07/850,751 --**US 5,621,126 (RE37,788E)**

Since the two-way obviousness test must be applied, it must also be shown that the claims of the cited, commonly owned patents could not have been filed in the instant application (e.g. are patentably distinct over claims 48-63 of this application). Specifically with regard to US 7,041,841 claims 12, 3, and 5 are directed toward symmetrically substituted (e.g. $x = 2$ or 4) Hf and Zr compounds useful for producing greater stereoregularity. This invention is a further development wherein a specific "improvement" catalyst compound is claimed. This improvement had not been identified at the time the invention in the instant application was filed and thus could not have been filed in the same application. Specifically the combination of Hf or Zr with x being 2 or 4 has an unexpected technical effect of enhanced stereoregularity. This is particularly true when one considers that titanium is usually the preferred species for the instant Mono-Cp catalysts. Therefore, claims 1, 2, 3, and 5 of US 7,041,841 could not have been filed in and are not obvious from the instant application. Thus the obviousness type double patenting rejection should be withdrawn.

With regard to US 5,612,126 and its reissue RE 37,788, claims 1, 2 and 6-9 of US 5,612,126 and claims 1-6 of RE 37,788 are directed toward specific catalyst compounds having the ability to produce ethylene alpha -olefin copolymers of high molecular weight with high levels of alpha-olefin comonomer incorporation and at high levels of catalyst productivity (see col. 1, lines 40-47 of '126). This invention is a further development wherein a specific "improvement" catalyst compound is claimed. This improvement had not been identified at the time the invention in the instant application was filed and thus could not have been filed in the same application. Specifically catalyst compounds having an R' group that is a 1° or 2° carbon atom have an unexpected technical effect of high levels of alpha-olefin comonomer incorporation at high molecular weights. Therefore, claims 1, 2 and 6-9 of US 5,612,126 and claims 1-6 of RE 37,788 could not have been filed in and are not obvious from the instant application. Thus the obviousness type double patenting rejection should be withdrawn.

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With regard US 6,617,466, claims 1-20 of US 6,617,466 are directed toward a process to the catalyst compound. US 6,617,466 claims priority to 07/533,245 which was subject to a restriction requirement as follows: Group I claims 1 to 6 and 14 to 17 which were directed to the catalyst compound and the process to make the catalyst compound and Group II, the catalyst system comprising the catalyst compound and an alumoxane. The claims in the instant application are directed toward the catalyst compound and a process of polymerization using the compound and an alumoxane. Applicant notes that the compound and the process to make the compound were originally grouped together in Group I. Therefore to facilitate prosecution, Applicant may be willing to submit a terminal disclaimer over US 6,617,466 in the event that the Examiner continues to believe that the double patenting rejection is proper.

Discussion of New Claims 83 to 121

New claims 83 to 121 are directed to a polymerization process using the catalyst compound claimed in claims 48 to 82 and an alumoxane. Similar claims were present in USSN 07/696,690 as claims 18-33. These claims were the subject of an interference, as noted above, that Applicant won. Those claims were cancelled out of the '690 application and are now presented here. Similar polymerization claims are presented in USSN 11/387,217. To facilitate prosecution Applicant will address prosecution issues regarding such claims raised in those applications.

First the polymerization process claims have been indicated as allowable over the prior art in USSN 387,217 and only stand rejected over five patents for obviousness type double patenting. Specifically, the Examiner in USSN 11/387,217 has stated "*No Prior art has been identified which anticipates or renders obvious the claimed method. The closest prior art is Tomotsu (US 5,420,089) and Campbell (US 5,045,017) each of which discloses titanium catalysts in combination with alumoxane or the polymerization of olefins. However, neither reference discloses or suggests the required bridge component which connects Cp with heteroatom ligands.*" However, the polymerization process claims do stand rejected for obviousness-type double patenting over claims 1-12 of US

5,227,440, claims 1-8 of US 5,420,217, claims 1-9 of US 5,504,169, claims 1-12 of US 5,516848, and claims 1-31 of US 5,844,055. In view of these obviousness-type double patenting rejections Applicant would like to note the following:

Claims 82-121 are directed to a polymerization process using a catalyst compound and alumoxane.

US 5,227,440, is directed toward a polymerization process using a supported catalyst compound and alumoxane. This is a further development wherein the polymerization process uses a catalyst which is supported on an inert support (all claims require an inert support). The successful support of these mono cyclopentadienyl catalysts was not easily accomplished until Applicant discovered a method thereafter permitting use of the supported catalyst polymerization processes, reducing metal residue in the final product and allowing said supported catalyst to be used in the gas -phase process. Variables included choice of support, particle size, surface area, contact method, recovery method, etc. for a given mono Cp catalyst used with alumoxane. All of these are evidence of unobviousness of the patent claims over the application claims 83 to 121. The lack of support disclosure in the earlier applications is also evidence of their unavailability and, therefore, unobviousness. Therefore, claims 1 to 12 of US 5,227,440 could not have been filed in and are not obvious from the instant application. Thus the obviousness type double patenting rejection should be withdrawn.

Claims 1-8 of US 5,420,217 are directed toward a process to produce amorphous polypropylene using a catalyst compound and an activator, where the catalyst compound is symmetrically substituted (e.g. $x= 2$ or 4). Note that at column 10, line 33-39, US'217 states "*As discussed further hereafter, by proper selection of the type and pattern R substituents for the cyclopentadienyl ligand in relationship to the type of R' substituent of the heteroatom ligand, the transition metal component for the catalyst system may be tailored to function in the catalyst system to produce low crystallinity poly-alpha-olefins or tactic amorphous poly-alpha-olefins.*" This invention is a further development wherein a specific "improvement" catalyst compound is claimed. This improvement had not been identified at the time the invention in the instant application was filed and thus

could not have been filed in the same application. Specifically selected symmetrically substituted catalyst compounds where x is 2 or 4 have an unexpected technical effect of producing an amorphous polymer with high molecular weights. This was unusual as at that time amorphous material was usually of low molecular weight. Therefore, claims 1-8 of US 5,420,217 could not have been filed in and are not obvious from the instant application. Thus the obviousness type double patenting rejection should be withdrawn.

US 5,504,169 is a CIP of US 5,420,217 with expanded activator coverage (specifically to include non-coordinating anion type activators). Like US 5,420,217 Claims 1-9 of US 5,504,169 are directed toward a process to produce amorphous polypropylene using a catalyst compound and an activator, where the catalyst compound is symmetrically substituted (e.g. x= 2 or 4). As described above for US 5,420,217 this was an improvement over the instant application and thus, claims 1-9 of US 5,420,217 could not have been filed in and are not obvious from the instant application. Therefore, the obviousness type double patenting rejection should be withdrawn.

Claims 1-12 of US 5,516,848 are directed toward a process for producing propylene based thermoplastic elastomer (TPE) like compositions using two different catalyst systems at the same time. The first catalyst system is a mono-Cp compound combined with an activator and the second catalyst system is a bis-Cp compound combined with an activator. This invention is a further development wherein two specific catalyst systems are selected to produce a specific kind of polymer (a TPE). This improvement had not been identified at the time the invention in the instant application was filed and thus could not have been filed in the same application. Therefore, claims 1-12 of US 5,516,848 could not have been filed in and are not obvious from the instant application. Thus the obviousness type double patenting rejection should be withdrawn.

Claims 1-31 of US 5,844,055 are directed to a process for producing certain copolymers of ethylene and a branched comonomer using a mono-Cp catalyst compound.

This invention is a further development wherein the specific kind of branched comonomer has profound effect on the crystalline behavior of the polymer (see column 8, line 25 of US 5,844,055). This improvement had not been identified at the time the invention in the instant application was filed (and in fact is not supported by the instant specification) and thus could not have been filed in the same application. Therefore, claims 1-31 of US 5,844,055 could not have been filed in and are not obvious from the instant application. Thus the obviousness type double patenting rejection should be withdrawn.

Furthermore, claims 18-33 (polymerization process claims) of USSN 676,690 were rejected for obviousness-type double patenting claims 1-13 of US 5,026,798. Applicant submits that the instantly claimed inventions are patentably distinct from claims 1-13 of US 5,026,798. Claims 1-13 of US 5,026,798 are directed towards a polymerization process for producing crystalline polyolefins having high molecular weight and narrow molecular weight distribution using a mono-Cp catalyst compound. Specifically, as stated at column 25, line 34-50 of US 5,026,798 "*By appropriate selection of (1) Group IVB transition metal component for use in the catalyst system; (2) the type and amount of alumoxane used; (3) the polymerization diluent type and volume; and (4) reaction temperature, one may tailor the product polymer to the weight average molecular weight value desired while still maintaining the molecular weight distribution at a value below about 4.0. The stereochemical control of the polymer formed is highly dependent on the exact structure of the transition metal component. Those transition metal components containing zirconium or hafnium (M=Zr or Hf) appear to have greater stereoregularity (fewer chain defects) than those containing titanium (M=Ti). By appropriate selection of the transition metal component of the catalyst system a wide variety of crystalline poly-.alpha.-olefins with differing stereochemical structure are possible.*" Such a process to produce a crystalline polymer is different from the instant process to produce polymer which may be crystalline, amorphous, etc. Note also that at column 16, line 52 of 5,026,798 the alpha-olefin is defined to have 3 to 20 carbon atoms. This does not include ethylene, which is well within the scope of the instant claims.

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Hence, claims 1-13 of US 5,026,798 represent a further development wherein a specific "improvement" with a different technical effect (e.g. obtaining stereoregular, crystalline polypropylene) is claimed. This improvement had not been identified at the time the invention in the instant application was filed and thus could not have been filed in the same application. Therefore, claims 1-13 of US 5,026,798 could not have been filed in and are not obvious from the instant application. Thus the obviousness type double patenting rejection should be withdrawn.

Relationship of Ti subgenus to Zr, Hf, Ti genus

Previously the Examiner has suggested that the genus of Zr, Hf and Ti is not separately patentable over the subgenus of Ti alone. In particular, in paragraph 2 of the office action dated 11/12/2004 the Examiner suggests that the lost count in interference 103, 819 (the titanium species case) precludes patenting of the genus case by the Applicant. Applicant respectfully disagrees and submits that the species and genus are separately patentable. In support of this position Applicant refers the Examiner to page 61 of the judgment from interference 102,954 (paper no. 681) where the Board of Appeals and Interferences clearly states that "*...a determination has already been made that the titanium subgenus is separately patentable from the zirconium. hafnium, titanium genus involved in this interference.*" Thus Applicant has reinstated claims to the full genus of zirconium. hafnium, and titanium.

New Claim 122

New claim 22 is similar to previous claim 27 and thus would likely be rejected under 35 USC § 103(a) as anticipated by Examples 2 to 12 of Campbell (US 5,045,517) which the Examiner suggests discloses a variety of Cp complexes, including Cp-Ti-phenoxy dichloride. The Examiner acknowledges that L is missing, but suggests that one would be motivated to add L because Campbell suggest addition of alcohols at Column 2, line 31-39. Applicant respectfully disagrees, and submits that Applicant's L group is not a separate molecule as the alcohol in Campbell is and thus is fundamentally different. Hence Applicant's claimed compound is not obvious from Campbell.

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Accordingly, Applicant respectfully requests the rejections to the claims be removed, and the claims, as amended be passed to allowance.

Please charge any deficiency in fees or credit any overpayments during the entire pendency of this case to Deposit Account No. 05-1712.

Date:

11/27/06

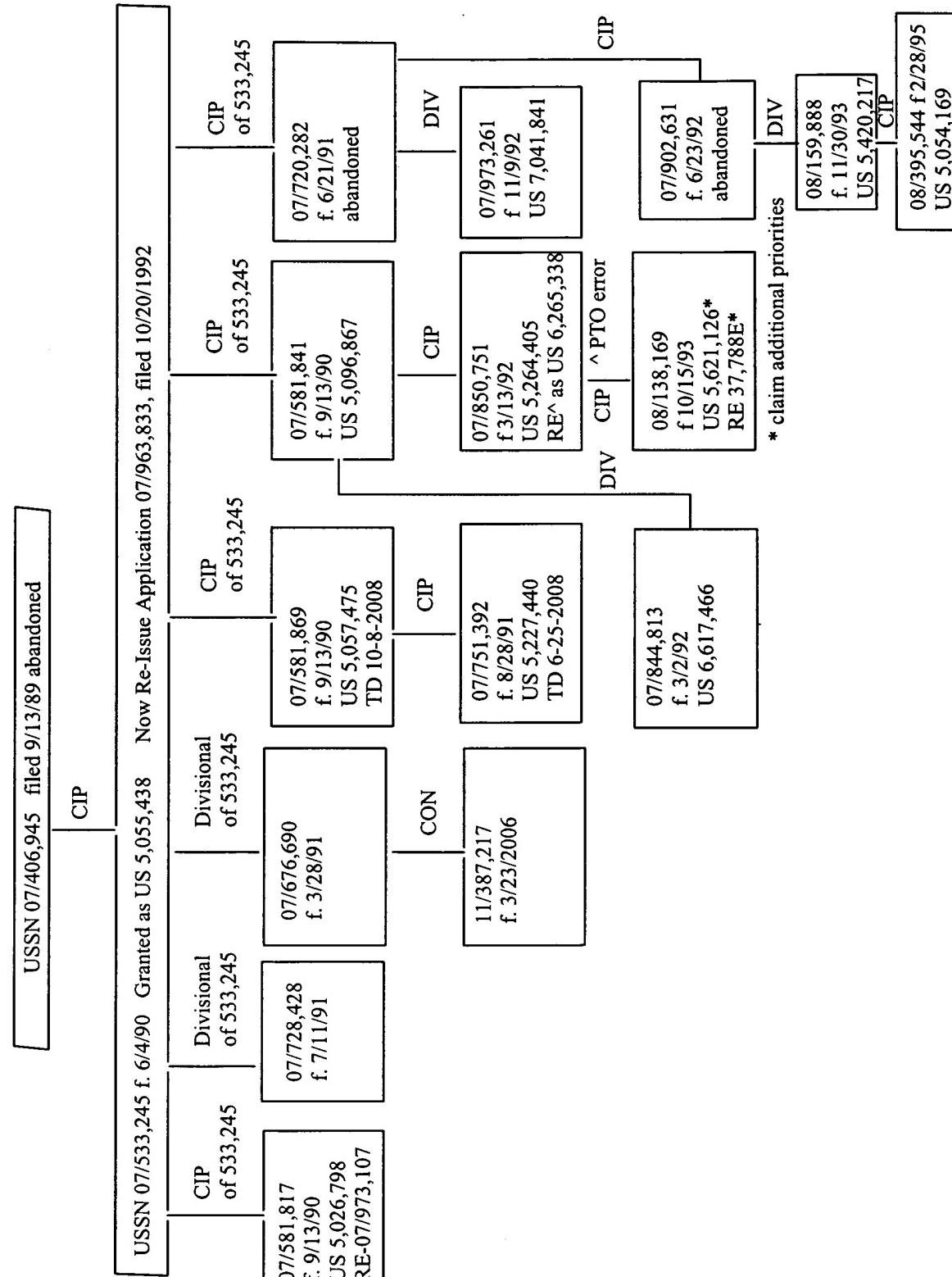
Respectfully submitted,



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All communications respecting this case should identify it by number and names of parties.



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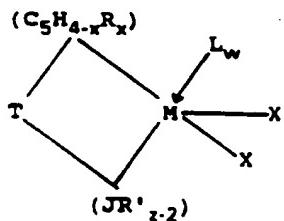
Applicant: Canich
Serial No.: 07/728,428
Filed: July 11, 1991
For: OLEFIN POLYMERIZATION
CATALYSTS

Accorded Benefit of: U.S.
S.Nos. 07/533,245, filed
06/04/90, now Patent No.
5,055,438, issued 10/08/91;
07/406,945, filed 09/13/89,
now abandoned

Pursuant to the APJ' decision on preliminary motion,
Interference No. 102,953 is redeclared by deleting count 1 and
substituting therefore new count 2 as follows:

Count 2

A compound having the general formula:



or a dimer thereof, wherein:

M is Zr, Hf, or Ti;

(C₅H_{4-x}R_x) is a cyclopentadienyl ring which is substituted with from zero to four substituent groups R, x is 0, 1, 2, 3, or 4 denoting the degree of substitution, and each substituent group R is independently a radical selected from the group consisting of C₁-C₂₀ hydrocarbyl radicals, substituted C₁-C₂₀ hydrocarbyl radicals wherein one or more hydrogen atoms is replaced by a halogen atom, C₁-C₂₀ hydrocarbyl-substituted metalloid radicals wherein the metalloid is selected from the group consisting of silicon and germanium, cyano, and halogen radicals, or (C₅H_{4-x}R_x) is a cyclopentadienyl ring in which two adjacent R groups are joined forming a C₄-C₂₀ ring to give a saturated or unsaturated polycyclic cyclopentadienyl ligand;

(JR'₂₋₂) is a heteroatom ligand in which "J" is an element with a coordination number of three from Group V-A or an element with a coordination number of two from Group VI-A of the Periodic Table of Elements, and R' is a radical selected from a group consisting of C₁-C₂₀ hydrocarbyl radicals, substituted C₁-C₂₀ hydrocarbyl radicals wherein one or more hydrogen atoms is replaced

Serial No. 728,428

by a halogen atom, and "z" is the coordination number of the element J;

X is, independently each occurrence, an anionic ligand group selected from the group consisting of hydride, halide, alkyl of up to 30 carbon atoms, alkoxy having up to a total of 30 carbon and oxygen atoms, cyanide, azide, acetylacetone, aryl having from 6 to 30 carbon atoms, aryloxy having a total of from 7 to 30 carbon and oxygen atoms, norbornyl and benzyl;

T is CR₂* or CR₂*CR₂* or SiR₂* or SiR₂*SiR₂* where R* is selected from the group consisting of hydrogen, C₁-C₂₀-alkyl, haloalkyl having up to a total of 20 carbon and halogen atoms, aryl having from 6 to 20 carbon atoms, and haloaryl having a total of from 7 to 20 carbon and halogen atoms;

L is a neutral Lewis base; and

w is a number from 0 to 3.

The claims of the parties designated as corresponding to this count are:

Canich: Claims 2, 4-6, 25, 26, 35-41 and 44-45.

Stevens et al.: Claims 1, 2, 49-56, 102, 103, 109 and 111-118.

Mary F. Downey

Mary F. Downey
Administrative Patent Judge
(703) 308-9821

MFD/raj

The opinion in support of the decision being entered today was not
written for publication and is not binding precedent of the Board.

Paper No. 578

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE BOARD OF PATENT APPEALS
AND INTERFERENCES

JAN 5 - 2001

FISH & NEAVE - PATENT DEPT
REFERRED TO
BY

DEC 29 2000

JO ANN M. CANICH
Junior Party¹

v.

JAMES C. STEVENS, FRANCIS J. TIMMERS,
DAVID R. WILSON, GREGORY F. SCHMIDT,
PETER N. NICKIAS, ROBERT K. ROSEN,
GEORGE W. KNIGHT and SHIH-YAW LAI
Senior Party²

Patent Interference No. 102,953³

Final Hearing: February 29, 2000

Before CAROFF, DOWNEY, and LORIN, Administrative Patent Judges.

LORIN, Administrative Patent Judge.

¹ Application 07/728,428, filed on July 11, 1991. Assigned to Exxon Chemical Patents, Inc., a company wholly owned by Exxon Corporation.

² Application 07/545,403, filed July 3, 1990. Assigned to The Dow Chemical Company.

³ This interference (102,953) is related to interferences 102,954, 102,955 and 103,067. The parties have agreed that, after they receive this decision, the Board will be informed if any issues remain to be resolved in the related interferences (see Paper No. 571).

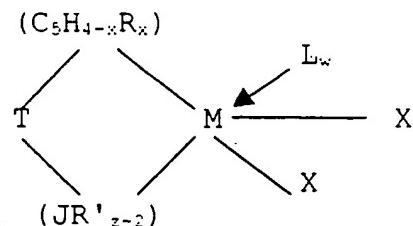
This is a Final Decision in the interference proceeding involving Canich Application 07/728,428, filed on July 11, 1991, and Stevens et al. (Stevens) Application 07/545,403, filed July 3, 1990.

Stevens has been accorded the benefit of an earlier filing date.⁴ Accordingly, Stevens is designated the senior party in this interference. 37 CFR §§ 1.657 and 1.601(m).

Count 2⁵, the sole count at issue, defines the interfering subject matter:⁶

Count 2

A compound having the general formula:



or a dimer thereof, wherein:

⁴ Canich's '428 application has been accorded the benefit of the September 13, 1989 and June 4, 1990 filing dates of earlier Canich applications 07/406,945 (now abandoned) and 07/533,245 (now U.S. Patent 5,055,438, issued October 8, 1991), respectively, and Stevens' '403 application has been accorded the benefit of the August 31, 1989 filing date of earlier Stevens' application 07/401,344 (now abandoned). See the Redeclaration of the interference (Paper No. 301); made pursuant to a decision on preliminary motion (Paper No. 299).

⁵ The Redeclaration Notice (Paper No. 301, p. 1) deleted original Count 1 and substituted Count 2.

⁶ 37 CFR § 1.601(f).

M is Zr, Hf, or Ti;

(C₅H_{4-x}R_x) is a cyclopentadienyl ring which is substituted with from zero to four substituent groups R, x is 0, 1, 2, 3, or 4 denoting the degree of substitution, and each substituent group R is independently a radical selected from the group consisting of C₁-C₂₀ hydrocaryl radicals, substituted C₁-C₂₀ hydrocarbyl radicals wherein one or more hydrogen atoms is replaced by a halogen atom, C₁-C₂₀ hydrocarbyl-substituted metalloid radicals wherein the metalloid is selected from the group consisting of silicon and germanium, cyano, and halogen radicals, or (C₅H_{4-x}R_x) is a cyclopentadienyl ring in which two adjacent R groups are joined forming a C₄-C₂₀ ring to give a saturated or unsaturated polycyclic cyclopentadienyl ligand;

(JR'_{z-2}) is a heteroatom ligand in which "J" is an element with a coordination number of three from Group V-A or an element with a coordination number of two from Group VI-A of the Periodic Table of Elements, and R' is a radical selected from a group consisting of C₁-C₂₀ hydrocarbyl radicals substituted C₁-C₂₀ hydrocarbyl radicals wherein one or more hydrogen atoms is replaced by a halogen atom, and "x" is the coordination number of the element J;

X is, independently each occurrence, an anionic ligand group selected from the group consisting of hydride, halide, alkyl of up to 30 carbon atoms, alkoxy having up to a total of 30 carbon and oxygen atoms, cyanide, azide, acetylacetone, aryl having from 6 to 30 carbon atoms, aryloxy having a total of from 7 to 30 carbon and oxygen atoms, norbornyl and benzyl;

T is CR₂^{*}, CR₂^{*}CR₂^{*}, SiR₂^{*} or SiR₂^{*}SiR₂^{*} where R^{*} is selected from the group consisting of hydrogen, C₁-C₂₀-alkyl, haloalkyl having up to a total of 20 carbon and halogen atoms, aryl having from 6 to 20 carbon atoms, and haloaryl having a total of from 7 to 20 carbon and halogen atoms;

L is a neutral Lewis base; and

w is a number from 0 to 3.

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The parties' claims which correspond to Count 2 are:⁷

- Canich's '428 application: claims 2, 4-6, 25, 26, 35-41, 44, 45; and,
- Stevens' '403 application: claims 1, 2, 49-54, 102, 103, 109 and 111-118.

The parties filed the following Briefs and Reply Briefs:

CB⁸ Canich Brief (redacted), filed November 1, 1999 (Paper No. 559)

SB Stevens Brief (redacted), filed January 21, 2000 (Paper No. 566⁹)

CRB Canich Reply Brief, filed February 2, 2000 (Paper No. 568).

Canich filed a Record and Exhibits (Paper No. 515, 516 and 523).¹⁰ Stevens filed a Record and Exhibits (Paper Nos. 494-497).¹¹ Both parties appeared at final hearing represented by counsel.

⁷ See the Redeclaration Notice (Paper No. 301, p. 3).

⁸ Hereinafter, the Briefs and Reply Briefs will be designated by these abbreviations followed by page number.

⁹ Another previous Brief (redacted) was filed on November 6, 1999 (Paper No. 563) but has been superseded by this one.

¹⁰ Hereinafter designated as CR and CX followed by page and exhibit number, respectively.

¹¹ Hereinafter designated as SR and SX followed by page and exhibit number, respectively.

The issues presented for our decision in the parties' briefs include Canich's priority case and Stevens' Motion 12. We will address the priority issue first, followed by a discussion of Stevens' Motion 12 and other issues raised by the parties in their briefs.¹²

No issue of no interference-in-fact has been raised.

PRIORITY

It is not the burden of the Board to scour the record, research any legal theory that comes to mind and serve generally as an advocate for a party. Compare Ernst Haas Studio, Inc. v. Palm Press, Inc., 164 F.3d 110, 112, 49 USPQ2d 1377, 1379 (2d Cir. 1999). Accordingly, in making our determination as to priority we have reviewed only those specific facts, issues¹³, and arguments of the parties relied upon in

¹² Matters not raised in a parties' brief are ordinarily regarded as abandoned. Photis v. Lunkenheimer, 225 USPQ 948 (Bd. Pat. Int. 1984).

¹³ The parties have provided the following Statements of the Issues regarding the question of priority:

Canich's Statement of the Issues (CB xviii)
Has Canich demonstrated that it is entitled to priority on Count 2 by a preponderance of the evidence based on actual reductions to practice prior to Stevens' earliest filing date of August 31, 1989?
Stevens' Statement of the Issues (SB xxxiii)

1. Has Canich proven by credible evidence that she actually made a metal compound of the count prior to Stevens' effective filing date?
2. Has Canich proven by credible evidence that a metal compound called for by the count was sufficiently tested to prove utility as a catalyst to polymerize olefins prior to Stevens' effective filing date?
3. Has Canich proven by credible evidence that she properly recognized the metal compound of the count and its utility prior to Stevens' effective filing date?

their briefs. See 37 CFR §§ 1.656(b)(5) and(b)(6).¹⁴

Stevens has been accorded senior party status and Canich has not challenged that status. Therefore, as the junior party, Canich has the burden of proof of establishing priority.

Stevens relies solely on a priority date of August 31, 1989 as its proof of a constructive reduction to practice (CB xxvii). Accordingly, Canich is under a burden of proof to establish a date of invention prior to August 31, 1989. Canich must establish that either it actually reduced to practice the invention of the count before August 31, 1989, or it first conceived the invention prior to that date and proceeded with reasonable diligence from a time just prior to the opponent entering the field toward a reduction to practice, either actual or constructive. 35 U.S.C. § 102(g). Haskell v. Colebourne, 671 F.2d 1362, 1365, 213 USPQ 192, 194 (CCPA 1982).

Canich¹⁵ seeks only to establish priority based on an actual reduction to practice of the invention of the count prior to August 31, 1989.

¹⁴ 37 CFR § 1.656(b)(5) requires:

[A] statement of the facts, in numbered paragraphs, relevant to the issues presented for decision with appropriate references to the record.

37 CFR § 1.656(b)(6) requires:

[A]n argument, which may be preceded by a summary, which shall contain the contentions of the party with respect to the issues it is raising for consideration at final hearing, and the reasons therefor, with citations to the cases, statutes, other authorities, and part of the record relied on.

¹⁵ Canich "seeks to establish by a preponderance of the evidence that at least a single species within the count of the invention claimed in the junior party's patent application was reduced to practice prior to the earliest filing date [i.e., August 31, 1989] that the senior party is entitled to the benefit of relative to the interference count" (CB 40).

In an interference proceeding, a party seeking to establish an actual reduction to practice must satisfy a two-prong test: (1) the party constructed an embodiment or performed a process that met every element of the interference count, and (2) the embodiment or process operated for its intended purpose.

Eaton v. Evans, 204 F.3d 1094, 1097, 53 USPQ2d 1696, 1698 (Fed. Cir. 2000).¹⁶ The first prong of the test requires a showing of both an actual preparation and a recognition of the embodiment of the count and the second prong of the test requires a showing of a recognition of a specific practical utility for that embodiment.¹⁷ Furthermore, an inventor's testimony must be corroborated by independent evidence in order to establish an actual reduction to practice. Knorr v. Pearson, 671 F.2d 1368, 1373, 213 USPQ 196, 200 (CCPA 1982), citing Reese v. Hurst, 661 F.2d 1222, 1225, 211 USPQ 936, 940 (CCPA 1981).

"With regard to the first prong, this Court's well-established precedent requires that the constructed embodiment ... include the precise elements recited in the count," Eaton v. Evans, 204 F.3d at 1097, 53 USPQ2d at 1698 (Fed. Cir. 2000). Canich urges that it "presented evidence concerning twelve reductions to practice before Stevens' earliest filing date of August 31, 1989" (CB 2) and focuses our attention on the actual reduction to

¹⁶ "[A] party cannot obviate the initial requirement that a constructed embodiment include every element of the count through evidence that the embodiment operated for its intended purpose, regardless of the quality of such evidence. Put simply, these are two distinct requirements and a party must satisfy each one to establish an actual reduction to practice." Eaton v. Evans, 53 USPQ2d 1696, 1699 (Fed. Cir. 2000).

¹⁷ "[R]eduction to practice requires a showing of three elements: (i) production of a composition of matter satisfying the limitations of the count, (ii) recognition of the composition of matter, and (iii) recognition of a specific practical utility for the composition." Estee Lauder v. L'Oreal S.A., 129 F.3d 588, 592, 44 USPQ2d 1610, 1613 (Fed. Cir. 1997), citing Standard Oil Co. (Indiana) v. Montedison, S.P.A., 494 F. Supp. 370, 206 USPQ 676, 689 (D.Del. 1980), aff'd, 664 F.2d 356, 212 USPQ 327 (3rd Cir. 1981), cert. denied, 456 U.S. 915 (1982).

practice of compound AZ (see CB 44-56).¹⁸ According to Canich (CB 44),

The compound that Canich made first was compound AZ, which has the chemical name: dimethylsilyl(tetramethylcyclopentadienyl)t-butylamido zirconium dichloride; and the chemical formula: Me₂Si(Me₄C₅)(N-t-butyl)ZrCl₂.

Canich provides a side-by-side comparison of AZ with the subject matter of Count 2 (CB 45-46). It is apparent to us, and Stevens does not dispute it, that AZ is a species within the scope of Count 2 and includes the precise elements recited in the count. Therefore, AZ could be relied upon as an actual reduction to practice of the invention in interference if, with respect to AZ, Canich can satisfy the two-prong test.

Reduction to practice is a legal question based on underlying factual determinations.

Fujikawa v. Wattanasin, 93 F.3d 1559, 1564, 39 USPQ2d 1895, 1899 (Fed. Cir. 1996).

Canich's burden with respect to the facts in trying to prove an actual reduction to practice of AZ is one of a preponderance of the evidence. 37 CFR § 1.657(b). Where "the burden is ... one of a preponderance of the evidence, then appellant's allegations of fact need be supported only by a preponderance of the evidence," Paivinen v. Sands, 339 F.2d 217, 222, 144 USPQ 1, 6 (CCPA 1964).

"[A] preponderance of the evidence ... standard ... only requires the fact finder 'to believe that the existence of a fact is more probable than its nonexistence before [he] may find in favor of the party who has the burden to persuade the [judge] of the fact's existence.' [citing In re Winship, 397 U.S. 358, 371-72 (1970)]."

¹⁸ For this reason, we view Canich's alleged reduction to practice of AZ as representative of all the alleged reductions to practice (e.g., of BZ, CZ, EZ, FZ, GZ, HZ, IZ, IZZ, JZ, and KZ - see CB Appendix).

Bosies v. Benedict, 27 F.3d 539, 542, 30 USPQ2d 1862, 1864 (Fed. Cir. 1994).¹⁹

Accordingly, in order for Canich to prevail, it must establish that, more probable than not, prior to August 31, 1989, the inventor or someone on the inventor's behalf (1) constructed AZ, by showing that (a) AZ was actually prepared and (b) that the compound that was prepared was recognized as AZ, and (2) recognized a specific practical utility for it. Canich's testimony in support thereof must be corroborated. We discuss each of these issues in turn: actual preparation, recognition, utility, and corroboration.

In determining whether Canich has met its burden of proving an actual reduction to practice with respect to AZ, we take into account Stevens' arguments. Stevens disputes that Canich actually made even one mono-Cp compound within the count (SB 99), and, even if Canich did, "Canich failed to perform the analytical studies necessary to establish the identity of a new compound of the type at issue here" (SB 73), and failed to show (SB 100) "the successful use of any compounds within the count as part of a catalyst system to polymerize olefins, the only utility Canich alleges" (SB 95). And furthermore, according to Stevens, Canich (SB 103) has failed to show corroboration.

¹⁹ Compare with the definition given in 5 CFR § 1201.56(c)(2) [revised January 1, 2000] for the preponderance of the evidence standard in administrative proceedings under the Merit Protection Systems Board [cited in Jackson v. Veterans Admin., 768 F.2d 1325, 1329 (Fed. Cir. 1985)]:

Preponderance of the evidence. The degree of relevant evidence that a reasonable person, considering the record as a whole, would accept as sufficient to find that a contested fact is more likely to be true than untrue.

See also St. Paul Fire & Marine Insurance Co. v. United States, 6 F.3d 763, 769 (Fed. Cir. 1993) which states that the "'preponderance of the evidence' formulation is the general burden assigned in civil cases for factual matters" and defines "preponderance of the evidence in civil actions to mean 'the greater weight of evidence, evidence which is more convincing than the evidence which is offered in opposition to it' [quoting Hale v. Department of Transportation, 772 F.2d 882, 885 (Fed. Cir. 1985)]."

Actual Preparation

To establish actual reduction to practice of AZ, Canich must prove "that the inventor actually prepared the composition and knew it would work." Mikus v. Wachtel [II], 542 F.2d 1157, 1159, 191 USPQ 571, 573 (CCPA 1976).

There is no dispute that, on four separate occasions, Canich prepared a compound that Canich alleges to be AZ. According to Canich (CB 22-25), preparation of a compound alleged to be AZ, was first begun on August 16, 1988 and completed on August 25-26, 1988. The compound was also prepared a second (completed September 23, 1988; CB 36), third (September 28-29, 1988; CB 37) and fourth (completed October 20, 1988; CB 38) time. Each of these preparations occurred prior to Stevens' August 31, 1989, priority date.

The August 16-26, 1988 preparation of AZ, which we take as representative, is discussed in detail in Canich's Brief at pp. 22-25, including the equipment used (CB 22), the starting materials (CB 23), and the reaction and precipitation conditions (CB 23-25). We reproduce the steps in the process of preparing AZ as they are described in Canich's Brief:

- "On August 16, 1988, Dr. Canich
 - reacted 230 mls. of degassed n-butyl-Li ... (purchased from Aldrich Chemical) with 40 mls of degassed NH₂-t-butyl ... (also purchased from Aldrich) in pentane using Schlenk techniques and
 - obtained LiHN-t-butyl [1] and the by-product butane." CB 23.
- "On August 17, 1988, Dr. Canich
 - purified the resultant compound by removing the solvent via vacuum in the drybox and
 - ran a proton NMR experiment ... [which NMR spectrum] is clean, showing peaks at the appropriate chemical shifts." CB 23.
- "Starting on August 19, 1988 and finishing on August 22, 1988, Dr. Turner and Mr. Matthew
 - reacted C₅Me₄O with LiAlH₄ in diethylether by adding 60.5 grams of C₅Me₄O dropwise to 235 cc of LiAlH₄ in 500 cc of diethylether using a dropping funnel.
 - The solution was refluxed for thirty minutes and

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- then hydrolyzed by adding 100 ml of water dropwise through the dropping funnel.
- The mixture was then cautiously added to a cold, aqueous 10% sulfuric acid solution.
- The ether layer was separated out, and
- the mixture was reduced to about 250 ml by evaporation.
- To this solution, with stirring was added dropwise 1 ml of concentrated sulfuric acid.
- The mixture was stirred for 3 hours and
- after additional work-up, Dr. Turner and Mr. Matthew obtained $C_5Me_4H_2$.
- The supporting NMR spectrum shows the appropriate peaks.
- Straightforwardly, $Me_4C_5H_2$ [2] was reacted with n-butyl-Li to obtain C_5Me_4HLi [3]." CB 23-24.
- "On August 23, 1988, Dr. Canich
 - reacted 10 grams of C_5Me_4HLi [3] ... [from Dr. Turner] with 10 ml Me_2SiCl_2 (purchased from Aldrich) ... in tetrahydrofuran ("THF") using Schlenk techniques to obtain 15.13 grams of $Me_2Si(C_5Me_4H)Cl$ [4]." CB 24.
- "On August 24, 1988, Dr. Canich
 - purified the resultant product by removing LiCl and
 - evaporating off the solvent." CB 24.
- "On the same day, Dr. Canich
 - obtained a proton NMR spectrum on the product [and]
 - recorded that the NMR 'looks good'." CB 24.
- "On August 24, 1988, still the same day, Dr. Canich
 - reacted 10 grams ... of $Me_2Si(C_5Me_4H)Cl$ [4] (from above) by adding it dropwise to 3.68 grams ... of NHLi-t-butyl [1] (from above) in about 100 ml of THF.
 - Dr. Canich removed the solvent,
 - precipitated out and
 - filtered off LiCl.
 - Dr. Canich added ether and
 - then 0.095 mole of MeLi dropwise to obtain 7.93 grams of $[Me_2Si(C_5Me_4)(N\text{-}t\text{-butyl})]Li_2$ [5]." CB 24.
- "On August 25, 1988, Dr. Canich
 - isolated the compound
 - and a proton NMR spectrum was taken on the product. ... The NMR had 'some bubbling', but otherwise shows the existence of the appropriate compound." CB 24.
- "On August 25, 1988, Dr. Canich
 - suspended 1.13 grams ... of $[Me_2Si(C_5Me_4)(N\text{-}t\text{-butyl})]Li_2$ [5] (from above) in about 100 ml of benzene and
 - slowly added 1.0g ... of $ZrCl_4$ (purchased from Cerac, Inc.).
 - After stirring overnight,
 - Dr. Canich filtered the mixture to dryness
 - and then extracted it with pentane and
 - placed the solution in the refrigerator at -40°C.
 - After filtration, Dr. Canich obtained 0.25g of $Me_2Si(C_5Me_4)(N\text{-}t\text{-butyl})ZrCl_2$ [6] (compound AZ as yellow solid)." CB 25.
- On August 26, 1988, Dr. Canich
 - recrystallized the product a second time (extraction) and obtained yellow crystals.
 - Two proton NMR spectra of the compound were taken.
 - The first was taken before the second recrystallization, and shows presence of the desired compound and two major peaks that should not be there.

- The second NMR spectrum was taken after second recrystallization ... and shows all the proper peaks." CB 25.
- On the same day that compound [6] was obtained (August 26, 1988), and
 - on the same laboratory notebook page,
 - Dr. Canich dissolved the 'remains from the side of the good flask' in toluene,
 - added 1 ml of methylalumoxane (obtained from a colleague (Dr. Welborn)) and
 - bubbled ethylene through the solution.
 - Dr. Canich observed the formation of polyethylene." CB 25.
- Compound [6] was used to prepare 'stock catalyst' solutions, which were used for several laboratory reactor polymerization experiments from September 1, 1988 through September 9, 1988." CB 25.

Canich (CB 25) maintains that the process described above was an actual preparation of the compound AZ. Stevens disagrees. Stevens (SB 62-63) takes the position that the result of this process, as well as the other attempts²⁰, is not AZ but other unintended compounds. Stevens argues that the process Canich used did not actually prepare AZ because it contained certain flaws in its execution:

... there are four basic flaws in the manner in which Canich attempted to synthesize the compounds of the count. These flaws apply to each alleged actual reduction to practice. First, Canich added reagents in the incorrect order. Second Canich added reagents in incorrect ratios. Third, Canich's synthesis experiments were heavily contaminated with impurities. And last, Canich failed to employ the correct analytical procedures necessary to establish the identity and purity of the metal complexes she prepared. ... these errors strongly point to the creation of unintended and unrecognized compounds such as bis-Cp metal complexes, rather than compounds corresponding to the count.

The four flaws in Canich's preparation of AZ that Stevens alleges to have occurred are the only flaws Stevens raises for our consideration. Accordingly, but for these four flaws, Stevens would appear to concede that the August 16-26, 1988 process described

²⁰ "As explained by Dr. Marks, there are four basic flaws in the manner in which Canich attempted to synthesize the compounds of the count. These flaws apply to each of the alleged actual reductions to practice," SB 62.

supra could in fact actually prepare AZ. We now analyze each of these flaws to see if the process described supra, more likely than not, prepared AZ rather than "unintended and unrecognized compounds such as bis-Cp metal complexes."

Stevens: Flaw #1 - Canich Added Reagents In The Incorrect Order

Stevens argues that Canich committed two errors involving adding reagents in the incorrect order.

With respect to the first error, our attention is directed to this step²¹ in the August 16-26, 1988, process (see supra):

- On August 24, 1988, still the same day, Dr. Canich
 - reacted 10 grams ... of $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4\text{H})\text{Cl}$ [4] (from above) by adding it dropwise to 3.68 grams ... of NHLi-t-butyl [1] (from above) in about 100 ml of THF.

According to Stevens (SB 64):

The first obvious synthesis mistake occurred in step 2.²² As Dr. Marks explained, Canich apparently added the silyl chloride compound dropwise to

²¹ We refer to the particular action taken during the actual August 16-26, 1988 preparation of AZ that corresponds to "step 2" of the synthetic route thought of by Canich (see footnote 14). That particular action occurred on August 24, 1988.

²² Stevens is referring to a synthesis route that Canich has stated it thought of in making AZ.

According to Canich (CB 7-8), Dr. Canich sought to make a zirconium analog of a compound made by Prof. John E. Bercaw (i.e., $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{ScCl}$). Around April 21, 1988, Dr. Canich thought of $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{ZrCl}_2$ (later known as compound AZ) and a way of making it. At the same time, Dr. Canich thought of a different but similar compound ($\text{Me}_2\text{Si}(\text{C}_5\text{H}_4)(\text{N-t-butyl})\text{ZrCl}_2$) and a synthesis route for this second compound - which was:

- (1) $\text{NaCp} + \text{Me}_2\text{SiCl}_2 \rightarrow \text{CpSiMe}_2\text{Cl} + \text{NaCl}$
- (2) $\text{CpSiMe}_2\text{Cl} + \text{LiHN-t-Bu} \rightarrow \text{CpSiMe}_2\text{HN-t-Bu} + \text{LiCl}$
- (3) $\text{CpSiMe}_2\text{HN-t-Bu} + 2 \text{Li-n-Bu} \rightarrow \text{LiCpSiMe}_2\text{NLi-t-Bu} + 2 \text{Bu}$
- (4) $\text{LiCpSiMe}_2\text{NLi-t-Bu} + \text{ZrCl}_4 \rightarrow \text{t-BuNSiMe}_2\text{CpZrCl}_2 + 2 \text{LiCl}$

"The route of synthesis that Dr. Canich thought of for compound AZ was the same, except that the starting compound was not NaCp , but was the lithium salt of tetramethylcyclopentadiene or LiHC_5Me_4 ." CB 8, fact 9.

a solution of the lithium amide compound at room temperature. This procedure was incorrect because it resulted in a local excess of the lithium amide salt. As explained by Dr. Marks, the following unintended compounds are likely to be created under such conditions: [see SB 65-67].

Stevens is arguing that Canich produced a local excess of the lithium amide salt when it dropwise added $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4\text{H})\text{Cl}$ to NHLi-t-butyl .

Canich disagrees. Canich does not dispute that a "local excess of the lithium amide salt" could be a problem in successfully obtaining AZ. However, according to Canich (CRB 110), the "local excess" condition was avoided as a result of "using a slowly dissolving ligand in dilute conditions".

Another technique used by chemists is to carefully select the solvent for the reaction and the amount of solvent relative to the reactant. If a solvent is chosen so that reactant A is fully dissolved in the solvent, but reactant B dissolves into the solvent only at a slow rate, a reaction between A and B will occur as slowly (or only as quickly) as reactant B dissolves into the solvent. ... A variation of this technique was used by Canich in conjunction with the synthesis of Compound AZ. Canich avoided the 'local excess' issue by following a known route for preparing the ligand and by using a slowly dissolving ligand in dilute conditions.

Canich is arguing that there was no "local excess" of the lithium amide salt as a result of the dropwise addition of the $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4\text{H})\text{Cl}$ to NHLi-t-butyl in 100ml of THF.

Accordingly, Stevens and Canich are taking opposite positions on whether a "local excess" of the lithium amide resulted from Canich's dropwise addition of the $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4\text{H})\text{Cl}$. A simple solution for resolving this argument would have been to conduct an experiment whereby $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4\text{H})\text{Cl}$ is dropwise added to NHLi-t-butyl in

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100ml of THF and see if a "local excess" of the lithium amide salt would or would not occur. However, the panel is not equipped to perform such an experiment. Accordingly, we rely on evidence submitted by the parties. In this regard, it is Stevens who raises the issue. Therefore, it is incumbent on Stevens to show that a "local excess" of lithium amide salt would more likely than not have been obtained when Canich dropwise added $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4\text{H})\text{Cl}$ to NHLi-t-butyl . That has not been done.

Stevens presents no experimental evidence demonstrating that Dr. Canich, in performing this step in the process, could not, as Canich asserts, have produced $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{Li}_2$. Instead, Stevens (SB 64, footnote 282) directs us to the Declaration of Dr. Tobin J. Marks (SR 162-295, see Paper No. 496) wherein Dr. Marks makes statements to the effect that "it is my opinion that other compounds ... would likely be formed in addition to the intended compound" (see, for example, SR 172, paragraph (43)).

Dr. Marks (SR 172- 173) states that

For step 5 (a) [the step at issue here] ... while the intended compound was $\text{Me}_2\text{Si}(\text{Me}_4\text{C}_5)(\text{N-t-butyl})\text{Li}_2$... it is my opinion that other compounds such as ... would likely be formed in addition to the intended compound. Additionally, compounds such as ... could also be present. Here ... the improper addition of the cyclopentadienyl compound dropwise to the solution of LiHN-t-Bu would have resulted in a large local stoichiometric excess of the lithium salt. Unintended compounds that are likely to form based upon the local stoichiometric excess of LiHN-t-Bu and its reaction with other reagents are shown below [Dr. Marks provides a diagram of the possible unintended compounds resulting from a "local excess"].

Dr. Marks attempts to cast a doubt on Canich's process by raising the possibility that other unintended compounds were produced when Canich performed the step of dropwise adding $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4\text{H})\text{Cl}$ to NHLi-t-butyl . However, Dr. Marks concludes rather than demonstrates that "the improper addition of the cyclopentadienyl compound dropwise to the solution of LiHN-t-Bu would have resulted in a large local stoichiometric excess of the lithium salt": Dr. Marks does not explain how Canich improperly added the cyclopentadienyl compound and why a "local excess" of the salt would have necessarily formed. There is no evidence that a dropwise addition of $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4\text{H})\text{Cl}$ to NHLi-t-butyl per se produces a "local excess" of lithium salt. Furthermore, it is mere speculation as to whether a "local excess" that might have been formed would result in only unintended compounds. First, there is no evidence that unequivocally shows that unintended compounds were actually made. Second, even if true and, as Dr. Marks submits, Canich obtained unintended compounds, this does not establish that Canich did not also obtain $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{Li}_2$. Both unintended compounds and $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{Li}_2$ could have been obtained and therefore, given that $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{Li}_2$ is an intermediate in AZ's preparation, it still would have been possible for AZ to have been prepared by Canich's August 16-26, 1988, process.

Accordingly, Stevens does not make a persuasive argument that an error was made when Canich performed the step of making $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{Li}_2$.

We reach the same conclusion with respect to the second alleged error raised by Stevens. The second alleged error, according to Stevens, involves the reaction conditions Canich employed in conducting the final step of synthesizing $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{ZrCl}_2$ (compound AZ). This step of Canich's August 16-26, 1988 process is at issue:

- "On August 25, 1988, Dr. Canich
 - suspended 1.13 grams ... of $[\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})]\text{Li}_2$ [5] (from above) in about 100 ml of benzene and
 - slowly added 1.0g ... of ZrCl_4 (purchased from Cerac, Inc.)."

According to Stevens (SB 67-71),

Canich made a second error in developing the protocol for Canich's step 4.²³ This is intended to add a single bidentate ligand (the "CpSiMe₂N-t-Bu" in Canich's chart) to each metal to produce the intended bridged metal complex as the dichloride (the "t-BuNSiMe₂CpZrCl₂" in Canich's chart). Once again, however, Canich selected reaction conditions unlikely to make the intended compound and, thus, this provides a second independent basis for the conclusion that Canich failed to prepare the intended metal complexes.

As Dr. Marks explained, appropriate conditions must be chosen in step 4 to avoid surrounding the metal Group 4 tetrachloride with a local stoichiometric excess of the dianion ligand. Otherwise, the likely result is the formation of unintended bis-Cp complexes. The reaction conditions chosen by Dr. Canich in all of the experiments at issue resulted in just such a local excess of the dianion ligand reagent. Because the dianion reagent was partially, or in some instances completely, dissolved in the solution prior to the slow addition of the metal tetrahalide, the most likely product of Dr. Canich's procedures would be bis-Cp complexes even if she used the correct mono-Cp ligand reagent. Indeed, Dr. Canich's selected conditions are exactly those employed when one intends to make bis-Cp complexes. SB 67-69.

Stevens is arguing that, during the final step of synthesizing $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{ZrCl}_2$, "the dianion reagent was partially, or in some instances completely, dissolved in

²³ See footnote 22.

the solution prior to the slow addition of the metal tetrahalide" and that this had the effect of producing "a local excess of the dianion ligand reagent". CB 69. Only if the dianion reagent was partially or completely dissolved could there be a local excess of dianion ligand reagent and only if there is a local excess of dianion ligand reagent could unintended compounds be formed.

While Canich does not dispute that either a "local excess" of dianion ligand might be produced if $[Me_2Si(C_5Me_4)(N-t\text{-butyl})]Li_2$ was partially/completely "dissolved" or that a "local excess" of dianion ligand would have been detrimental to the production of AZ, Canich (CRB 114-115) disputes that $[Me_2Si(C_5Me_4)(N-t\text{-butyl})]Li_2$ was "dissolved". According to Canich, the $[Me_2Si(C_5Me_4)(N-t\text{-butyl})]Li_2$ was "suspended", not "dissolved", thereby precluding both the formation of a "local excess" of dianion ligand and resulting nonintended compounds.

Stevens says that "appropriate conditions must be chosen in step 4 to avoid surrounding the metal Group 4 tetrachloride with a local stoichiometric excess of the dianion ligand" Stevens goes on to say that Dr. Canich chose the wrong conditions. However, , Dr. Canich used the technique of carefully choosing her solvent and its amount so that the dianion ligand was "suspended" in the solvent "Suspended" means not dissolved. Thus, as Dr. Canich added the $ZrCl_4$, the $ZrCl_4$ was able to react only with the dissolved dianion ligand, which was a very small amount. Thus, it was not possible to have a "local excess" of the dianion reagent as Stevens asserts. CRB 114-115.

As with the first alleged error, there is no experimental evidence that would help the panel resolve the issue. Stevens provides no experimental evidence showing that Canich

"dissolved" rather than "suspended" $[\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})]\text{Li}_2$. We have no concrete evidence showing the formation of a "local excess" under the reaction conditions used by Canich; that is, whenever one "suspends" 1.13 grams of $[\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})]\text{Li}_2$ in about 100 ml of benzene.

Instead, Stevens (SB 69; footnotes 300 and 301) directs our attention to the following Canich exhibits (Paper No. 497). We quote the relevant language from each of the exhibits Stevens cites:

- CX 1276 at 41: "suspended in ... Et_2O "
- CX 1373 at 139²⁴: "suspended in ~100 ml benzene"
- CX 1373 at 145: "added to ~ 100 ml toluene"
- CX 1373 at 149: "mixed into ~100 ml ET_2O "
- CX 1373 at 161: "mixed into ~100 ml ET_2O "
- CX 1400 at 3: "suspended in ~ 100 ml Et_2O "
- CX 1400 at 4: "suspended in ~ 100 ml Et_2O "
- CX 1400 at 8: "suspended (largely dissolved) in ~ 100 ml Et_2O "
- CX 1400 at 9: "suspended (largely dissolved) in ~ 100 ml Et_2O "
- CX 1400 at 29: "dissolved suspended in ~ 50 ml Et_2O "
- CX 1400 at 34: "began to dissolve/become suspended in the Et_2O "
- CX 1400 at 35: "suspended in ~ 30 ml Et_2O "
- CX 1400 at 37: "suspended in ~ 100 ml Et_2O "
- CX 1400 at 40: "suspended ... in ~ 100 ml Et_2O "
- CX 1400 at 42: "suspended in ~ 50 ml Et_2O "
- CX 1400 at 43: "suspended in ~ 50 ml Et_2O "
- CX 1400 at 55: "suspended in Et_2O (~50 ml)"
- CX 1400 at 56: "suspended in Et_2O (~50 ml)"
- CX 1400 at 60: "suspended in ~ 30 ml Et_2O "
- CX 1400 at 61: "suspended in ~ 50 ml Et_2O "
- CX 1400 at 62: "suspended in 40 ml Et_2O "
- CX 1400 at 63: "suspended in Et_2O (~50 ml)"

²⁴ The entire sentence reads:

$\text{LiMe}_4\text{CpSiMe}_2\text{N}(\text{t-Bu})\text{Li}$ (1.13g = 3.80 4.29 mole) was suspended in ~ 100 ml benzene. 1.0g (4.29 mole) ZrCl_4 was slowly added. The mixture turned a dark yellow upon addition.

- CX 1400 at 71: "suspended in ~120 ml Et₂O"
- CX 1400 at 72: "suspended in ~120 ml Et₂O"
- CX 1276 at 39: "diluted in approx 75ml of the solvent, Et₂O"
- CX 1276 at 44: "dissolved in ~ 75 ml of the solvent Et₂O"
- CX 1276 at 45: "dissolved in ~ 75 ml of the solvent Et₂O"

Only exhibit CX 1373 mentions benzene. Accordingly, only CX 1373 corresponds to the step in Canich's August 16-26, 1988 process that is at issue here. However, nowhere in the CX 1373 does it state that the ligand was "dissolved". It states, as Canich argues, that the ligand was "suspended". There is nothing in this exhibit to suggest that the "suspended" ligand was in fact "dissolved" in benzene.

The other exhibits (e.g., CX 1400) involve solvents other than benzene and do not suggest that Canich in fact dissolved the ligand. Only CX 1276 at 44-45 unequivocally indicates that Canich "dissolved" the ligand. However, in that preparation, 4 g of ligand were "dissolved" in ~ 75 ml of Et₂O. This compares with the 1.13 grams of ligand that was added to 100 ml of benzene in Canich's August 16-26, 1988, process. It is not at all clear that an analogy can be made to the August 16-26, 1988 process based on the ligand's tendency to dissolve or suspend in the Et₂O. Given that Stevens provides no explanation as to why one would view Et₂O and benzene as equivalent alternatives, comparing these two syntheses, which use different amounts of ligand and solvent as well as different types of solvent, is tantamount to comparing apples and oranges. Accordingly, these exhibits do not provide the necessary support for the argument Stevens has made that Canich "dissolved", rather than "suspended", the ligand when it conducted the August 16-26, 1988

preparation. Therefore, Stevens' argument that Canich produced a "local excess" of dianion ligand is based on mere speculation. Accordingly, Stevens' does not provide a persuasive argument that Canich formed unintended compounds rather than AZ.

Stevens makes two other arguments with respect to this second error.

First (SB 68), relying on Dr. Marks (SR 179-180), Stevens argues that "appropriate conditions must be chosen in step 4 to avoid surrounding the metal Group 4 tetrachloride with a local stoichiometric excess of the dianion ligand." SB 68. In that regard, we point out that all that Dr. Marks (SR 179) states is that

Specifically, for step 7(a) of the chart in ¶36 [SR 168], above, while the intended compound was AZ, $\text{Me}_2\text{Si}(\text{Me}_4\text{C}_5)(t\text{-BuN})\text{ZrCl}_2$, ... it is my opinion that many unintended bis(cyclopentadienyl) compounds would, in fact, be the products formed from this reaction procedure. The reason for this is that Canich improperly added the ZrCl_4 to the solution of the alleged dianion reagent which resulted in a large local stoichiometric excess of this reagent. This large local stoichiometric excess of ligand would result in the formation of many unintended bridging ligand complexes.

There is nothing here but opinion and conclusory statements. There is no concrete evidence to persuade us that Canich produced a "large local stoichiometric excess of ligand."

Second, Stevens (SB 70) argues that "the formation of bis-Cp zirconium ... metal complexes, following the synthesis procedures used by Dr. Canich, has been fully documented in the literature." One example is, according to Stevens, Du Plooy et al.²⁵

²⁵ Karen E. Du Plooy, Ulrich Moll, Sigrid Wocadlo, Werner Massa, and Jun Okuda, "Coordination Properties of Novel Tridentate Cyclopentadienyl Ligands in Titanium and Zirconium Complexes," Organometallics 1995, 14, 3129-3131. See SX 1570.

However, there is no indication in this reference of a step of "suspending" $[\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})]\text{Li}_2$ in benzene. We therefore cannot conclude, as Stevens has, that this step in Canich's process could produce only unintended bis-Cp complexes and not AZ.

Stevens argues that Canich committed errors involving adding reagents in the incorrect order in two different steps in Canich's preparation of AZ. For the foregoing reasons, we are not persuaded that the evidence shows that Canich more likely than not committed these errors, thereby jeopardizing its ability to prepare AZ when it conducted the steps described supra for its August 16-26, 1988 preparation.

Stevens: Flaw #2 - Canich Added Reagents In Incorrect Ratios

Stevens argues that Canich used starting materials in the wrong ratios. According to Stevens (SB 71-71):

Another problem with Canich's technique results from the fact that the starting compounds used in Canich's syntheses can combine in the intended way, or in unintended ways, depending on the ratios of the starting materials employed. The use of improper ratios results in the formation of impurities in each of the synthetic steps. These impurities can thereafter combine with reagents used in subsequent steps to produce cascades of unintended metal complexes. An example of Dr. Canich's improper procedure includes the method that she used with lithium alkyls. Dr. Canich explained that lithium alkyl solution was simply added until the solution turned brown. As explained by Dr. Marks, Dr. Canich's technique resulted in the presence of a large excesses of methyl lithium. Because methyl lithium is a known reducing agent, the excess of methyl lithium likely resulted in the reduction of the metal complexes present. This too would have resulted in the formation of unintended metal complexes that are outside the scope of the count. Again, the application of appropriate analytical techniques would have revealed the

mistake and changes in procedure could have been implemented.

In arguing that Canich used starting materials in the wrong ratios, it is unclear which starting materials Stevens is referring to. Stevens refers to Dr. Canich's use of lithium alkyls but it is unclear how this applies to the August 16-26, 1988 preparation that is being analyzed here. Stevens does not explain what the proper ratios for this or any other starting material should have been. Accordingly, it is impossible to determine if this alleged error was committed during the August 16-26, 1988 preparation.

We note Stevens' argument that Dr. Canich used an improper technique when she added a lithium alkyl solution. However, Stevens never explains in what way the technique was improper. Stevens refers to statements made by Dr. Marks (SR 166), for example:

In addition, many of Canich's synthetic experiments specifically indicate that a stoichiometric excess of lithium alkyl reagent was employed. Lithium alkyls are known to reduce the oxidation state of Group 4 metals. Thus, stoichiometrically excess lithium alkyls are likely to result in the formation of greater than expected amounts of paramagnetic species, such as Hf(III), Ti(III) or Zr(III) compounds.

However, Dr. Marks does not actually state that Dr. Canich's technique was improper. Dr. Marks states that a "stoichiometric excess of lithium alkyl reagent" was employed and then speculates that unintended compounds may have been produced. There is no evidence that Dr. Canich's technique was improper per se and that, as a result, Canich's August 16-26, 1988 preparation could not have synthesized AZ but rather could have synthesized only unintended compounds.

Stevens argues that Canich committed an error involving reagents in the wrong ratios. For the foregoing reasons, we are not persuaded that the evidence shows that Canich more likely than not committed this error, thereby jeopardizing its ability to prepare AZ when it conducted the steps described supra for its August 16-26, 1988 preparation.

Stevens: Flaw #3 - Canich's Synthesis Experiments Were Heavily Contaminated

Stevens argues that Canich's synthesis experiments were contaminated and as a result, there is no guarantee that Canich actually prepared, for example, AZ. According to Stevens (SB72-73):

In attempting to synthesize the metal complexes required here, most of the steps must be conducted in an inert atmosphere, such as that provided by an efficient drybox. The evidence showed that Canich's dryboxes were contaminated with oxygen, water and other impurities. Indeed, Dr. Burkhardt, a scientist at the same Exxon research facility and one of Canich's experts here, identified not only oxygen and water, but also carbon monoxide, carbon dioxide, sulfur compounds and acetylene as possible impurities.

The evidence on which Stevens relies in support of its argument is presented in Stevens' Brief at SB 75-82. According to Stevens:

- Most of Canich's synthesis work took place in Dr. Canich's drybox (SB 77 ¶142; relying on CR 7553-68);
- "The mono-Cp metal complexes at issue here are known to be very unstable in the presence of contaminants," (SB 75 ¶138, relying on SR 150-51, SR 8672-75, 8712-13, CR 10272);
- "The evidence shows that Canich's drybox was contaminated" (SB 79 ¶146; relying on SR 248-54); "Indeed, the evidence points to the fact that Canich's dryboxes were highly contaminated," (SB 75 ¶138, relying on CR 1789-93; SR 1503-07; CR 1975-76; SR 272-73);
 - "For example, the evidence showed that the inlet and exit ports for the inert gas source were both located on the back wall of the drybox, at the same height,

- separated by only two feet. This situation creates a 'short circuit' problem in the drybox, a problem that leads to increased contamination from, for example, leaks in the drybox," (SB 79 ¶145; relying on CX 597; SX 1561);
- "Canich noted bubbling in several experiments in which a solution was prepared of a sensitive complex, pointing to the reaction of the compound with water and the release of a gas such as methane," (SB 79 ¶146; relying on CR 16-18; CX 1373; SR 172-74);
 - "Canich even recorded in her notebook that a compound stored in a sealed vessel in her drybox decomposed visibly within an hour," (SB 79 ¶146; relying on CR 1975-76; CX 1400);
 - "Canich also noted the presence of impurities in her NMR spectra," (SB 79 ¶146; relying on CR 1783-84; 1908-09; 1963-69; 8357-61; 8411-26; 3056; 3103-04; 3124; 3131-32; SX 1568);
 - "Canich also admitted that her drybox was 'heavily full of ether,'" (SB 79 ¶146; relying on CR 1789-93; SR 1503-07);
 - "Canich repeatedly used chlorinated solvents and reagents in her drybox," (SB 80 ¶148; relying on CR 4097-4102);
 - "Volatile compounds can also be introduced ... In recognition of this problem, researchers keep reagents in Sure/Seal™ bottles ... Canich apparently used ordinary bottles for this reagent, rather than Sure/Seal™ bottles, resulting in certain contamination of her drybox atmosphere," (SB 77 ¶141; relying on SX 1589);
 - "contamination by laboratory air results in much higher levels of contamination by oxygen than by water. Canich's own expert, Dr. Schrock, identified the possible contamination levels as sufficient to 'rapidly destroy' a 5 mmol sample (about 2 grams) of the metal complex at issue," (SB 76 ¶139; relying on CR 4098-12);
 - "Volatile components can enter the drybox ... by diffusion ... leaks ... cracks ... opening the inner door...," (SB 76 ¶140; relying on SR 251-52, CR 4097-98);
 - "Canich ... did not take the appropriate measures to ensure that her drybox atmosphere was free of contaminants that would have destroyed any mono-Cp compound that was present," (SB 75 ¶138, relying on SB 76-82 ¶139-51);
 - "Canich alleges that the purification bed called a "Dri-Train™" would have removed all contaminants from her drybox atmosphere. However ... purification beds [must] be periodically regenerated and replaced. The need to regenerate or replace the purification beds can be determined by monitoring the levels of contamination [especially, according to Stevens, that of oxygen]," (SB 80 ¶138);
 - "Canich ... did not monitor the oxygen levels in her drybox. At best, Canich monitored the water levels. ... However, simply monitoring contamination by water is an insufficient check on the amount of oxygen contamination in the drybox

atmosphere because, as stated above, oxygen is a more common contaminant ... and the capacity for its removal is lower," (SB 81 ¶149, relying on CR 7548-49, 7577-78, 7803-04, 4032, 7595, 7599, 4094-98; SR 3960-62);

Canich (CRB 117 and 36-44) responds by arguing that "Dr. Canich and her laboratory assistants followed good laboratory practices when dealing with the air and moisture sensitive compounds at issue here" (CRB 36). According to Canich (see, e.g., CRB 41), Stevens' assertions that Canich's dryboxes were contaminated are mere speculation. According to Canich (CRB 43-44),

The Exxon dryboxes were always properly operated, either in a recirculating mode through Dri-trains™ or in a continuous purge mode using high purity nitrogen. ... almost all of Canich's dryboxes were equipped with moisture meters, some were equipped with oxygen meters, and in every case chemical tests for oxygen and moisture were standard procedure when instrumental methods were not used. ... All Exxon dryboxes that used high purity nitrogen obtained it from the same source ... Therefore, any conceivable oxygen contamination from nitrogen is effectively monitored by a single oxygen meter on the nitrogen supply or in a drybox using nitrogen purge. Any conceivable oxygen contamination from atmospheric intrusion into a drybox would be accompanied by contamination by atmospheric moisture. Therefore, any conceivable oxygen contamination from the ambient atmosphere is effectively monitored by monitoring the moisture level in each drybox. As a result, effective monitoring for water and oxygen contamination in Exxon's dryboxes would require at a minimum the monitoring for oxygen of the nitrogen supply at a single point and the monitoring of moisture in each drybox. In actual fact, the Exxon experimenters went beyond this minimum to monitor for oxygen and moisture in each drybox by instrumental methods or by chemical methods, or by both.

Accordingly, the parties take divergent views with respect to whether Canich, in performing, for instance, the August 16-26, 1988 synthesis, used a contaminated drybox.

Both parties appear to agree, however, that a contaminated drybox could hamper the synthesis of AZ.

None of the evidence that Stevens presents establishes that Canich used dryboxes so contaminated that no AZ could possibly be prepared. There is disagreement as to whether any contamination existed within the dryboxes. In this regard, Stevens has not provided any experimental evidence of the levels of contamination that would result from each of the problems that Canich might have encountered. Accordingly, Stevens' assertions that Canich conducted experiments in a contaminated drybox is based on speculation. But even if Stevens were correct and a conclusion of contamination could be made based on, for example, the Canich evidence (SB 79 ¶146) showing that Dr. Canich herself indicated the presence of impurities, the question is not whether there was contamination per se but whether there was contamination sufficient to prevent the formation of compound AZ. That has not been shown.

The question is whether Canich actually prepared AZ. In answering the first prong of the test for determining an actual reduction to practice, Canich must show by a preponderance of the evidence that AZ was actually prepared. Canich cannot show by a preponderance of the evidence that AZ was actually prepared if, as Stevens urges, the drybox was contaminated to a level that prevented AZ from being made. Accordingly, the question of contamination becomes important only if we know, first, the actual amount of contamination in the drybox that Canich used and, second, the maximum allowable level of

contamination that would have to be present to prevent AZ from being synthesized. Only by comparing the actual to the maximum allowable levels of contamination can one determine whether Canich actually prepared AZ in light of any contamination of the drybox. However, there is no evidence of the specific level of contamination that was present in Canich's drybox. There is also no evidence of the level of contamination that would have been necessary to prevent AZ from being formed. Since this information has not been provided, it is impossible to determine whether, when Canich performed its August 16-26, 1988 preparation, Dr. Canich used her drybox in a manner such that Canich was prevented from making AZ.

There is also the matter of whether Dr. Canich's use of a "Dri-TrainTM" removed all contaminants from her drybox atmosphere. Stevens does not dispute that Canich used a "Dri-TrainTM" and does not show that this is either improper or insufficient. Stevens explains that purification beds must be periodically regenerated and replaced to be effective and that monitoring the levels of contamination must still be performed. But Stevens does not demonstrate that Canich did not do so. Stevens is merely pointing out the difficulties surrounding the purification of dryboxes without spelling out in what manner Canich failed to do so. Accordingly, we are not persuaded that whatever atmospheric contamination did exist, Canich did not properly and satisfactorily employ a Dri-TrainTM to remove it.

Stevens argues that Canich's synthesis experiments were contaminated and, as a result, there is no guarantee that Canich actually prepared AZ. For the foregoing reasons,

we are not persuaded that the evidence shows that Canich more likely than not contaminated the drybox to such a level that it jeopardized its ability to prepare AZ when it conducted the steps described supra for its August 16-26, 1988 preparation.

Stevens: Flaw #4 - Canich Failed To Employ Correct Analytical Procedures

The last alleged flaw, according to Stevens (SB 62-63), is that "Canich failed to employ the correct analytical procedures necessary to establish the identity and purity of the metal complexes she prepared." In particular, Stevens (SB 73-74) argues that

As already discussed at length, Canich failed to perform the analytical studies necessary to establish the identity of a new compound of the type at issue here. In particular, Canich never bothered to conduct routine tests for elemental composition. As a consequence, Canich's analytical data is consistent with the bis-Cp metal complexes that Dr. Marks testified were the likely products of Canich's experiments.

Moreover, as discussed earlier, the quality of Canich's NMR spectra is very poor. Dr. Canich admittedly never bothered to adjust the NMR equipment or to properly reference and maintain the data that were obtained. These problems undermine her ability to rely on the spectra, particularly the observed chemical shifts and integration values to help confirm the identity of the compounds she prepared.

In sum, Canich did not obtain the quality and quantity of analytical data necessary for someone skilled in the art to establish the identity of whatever metal complexes she made. No amount of flawed data (of the type Canich relies upon here) will ever support Canich's asserted synthesis results.

This alleged flaw does not go to the question of whether Canich actually prepared AZ, which is at issue in this section, but to whether Canich recognized or appreciated that the compound it prepared was AZ. Accordingly, we do not address it here. It is more properly

addressed in the following section where we review whether Canich has satisfied its burden, by a preponderance of the evidence, to show it recognized or appreciated AZ when it was prepared.

But for the alleged flaws, Stevens does not appear to question the process for actually preparing AZ that Canich describes in its brief. For the foregoing reasons, we are persuaded that the preponderance of the evidence shows that Canich more likely than not actually prepared AZ when, for example, it conducted the steps described supra for its August 16-26, 1988 preparation.

Recognition or Appreciation

In order to satisfy the first prong of the test for establishing an actual reduction to practice Canich must also establish that it recognized or appreciated AZ when it was prepared. Accordingly, the issue here is whether the preponderance of the evidence shows that Canich confirmed that the product it had obtained was the product they identified as AZ.

Canich asserts that they did enough analytical testing to convince the inventor and others skilled in the art that the inventor in fact prepared the compounds she intended to make. CB 47-48. That testing included performing a proton NMR on August 26, 1988 (CB 25) subsequent to the first synthesis [i.e., the August 16-26, 1988 preparation discussed supra], as well as NMR analyses on the other three synthesized compounds (CB Tab 3, pp.

1-2; pp. 7-8), and x-ray crystallographic analyses conducted by Dr. Cynthia S. Day. Day's crystallographic analysis was conducted subsequent to the fourth synthesis. On March 14, 1989, Dr. Day orally reported the preliminary results of the analysis to Dr. Canich. Final results were reported in August of 1989 (CB Tab 3, p. 8). Adding to this, Canich relies on NMR spectra for AZ² - a dimethylated version of AZ - obtained from the derivatization work of Dr. Turner, conducted independently of inventor Canich (CB Tab 3, pp. 12), to show that the compound on which it was based had to be AZ. Prior work on similar compounds and subsequent work by others showing inventor Canich's spectra as being consistent with AZ are also relied upon. In conclusion, Canich states that "taken as a whole (including the recently published data, which confirms Canich's data) it is convincing that Dr. Canich in fact made Compound AZ" (CB 50). We agree.

Stevens (SB 11) argues that

One of the most glaring deficiencies of Canich's case is Dr. Canich's failure to use adequate analytical techniques to identify reagents, intermediates or products in her experiments. At the top of the list is Canich's failure to perform any recognized and readily available test to determine the elemental composition of the samples through appropriate elemental analysis techniques. These techniques, which include combustion analysis and mass spectrometry, indicate the percentage elemental composition of the compound and, in the case of mass spectrometry, the molecular weight of the compound. These two simple, readily available elemental analysis techniques, in combination with properly performed NMR spectroscopy and x-ray crystallography, would have distinguished between the intended and unintended compounds in every one of Dr. Canich's experiments. Yet, she failed to use these techniques in any of her experiments. In addition, Dr. Canich's demonstrated lack of basic competency in record keeping and careless approach to the use of NMR spectroscopy and other analytical techniques makes Canich's evidentiary record of little or no value. In any

event, even if the NMR tests were properly carried out, they could not, without elemental analysis, prove the existence of a metal complex within the scope of the count. The basic defect applies to all of Canich's alleged twelve reductions to practice and alone defeats Canich's attempt to prove a reduction to practice.

Accordingly, Stevens is presenting two arguments: A) that Canich did not perform an elemental analysis and accordingly could not have known whether the sample it obtained was a compound within the scope of the count, and B) Canich's record keeping is such that there is little or no evidence to substantiate Canich's claim that it in fact made a compound within the scope of the count. Because Canich failed to perform an elemental analysis and failed to properly document the results, according to Stevens, Canich did not recognize or appreciate that it prepared a compound within the scope of the count. We review each of these two arguments in turn.

A) Analytical Testing

Focusing on the August 16-26, 1988 preparation of AZ discussed supra, Canich, in meeting its burden of establishing that it recognized and appreciated that it prepared AZ, relies on two NMR spectra. This is reflected in the following activity of August 26, 1988, reproduced from Canich's Brief:

- On August 26, 1988, Dr. Canich
 - recrystallized the product a second time (extraction) and obtained yellow crystals.
 - Two proton NMR spectra of the compound were taken.
 - The first was taken before the second recrystallization, and shows presence of the desired compound and two major peaks that should not be there.
 - The second NMR spectrum was taken after second recrystallization ... and shows all the proper peaks." CB 25.

With respect to the NMR spectra, Stevens (SB 11-38) argues that (1) by employing that technique, Canich was not in accord with standard Exxon procedures and (2) such a technique cannot distinguish between compounds having similar structure.

(1) Elemental Analysis

Stevens (SB 11) asserts that "Canich failed to conduct routine analytical testing necessary to establish what was synthesized." Stevens urges that Canich should have employed elemental analysis since this was a standard technique for identifying new metal complexes.

Stevens directs our attention to the testimony of Dr. Burkhardt, a Senior Staff Chemist at Exxon (SB 13), and Dr. Floyd of Exxon's Materials Characterization Laboratory (SB 14). Dr. Burkhardt (SB 14) testified that a schematic representation of a compound based on NMR spectra may be incorrect. Dr. Burkhardt does not state, as Stevens appears to suggest (SB 13), that NMR spectra are "inadequate" to characterize compounds such as AZ. Depending on other circumstantial evidence, it is quite possible that a schematic representation of a compound based on NMR spectra could be correct. That is apparently the situation here. Canich (CRB 48) is not relying on the NMR spectra as the sole evidence proving that AZ was prepared. For example, Canich (CRB 48) points out that Dr. Canich "was not starting from scratch," basing the resulting compound as well as its method of preparation on the scandium compounds previously prepared by Dr. Bercaw. This is buttressed by the clear similarity between what Dr. Bercaw prepared -

$\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{ScCl}$ - and what Dr. Canich intended to prepare - $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{ZrCl}_2$. Regarding Dr. Floyd, the testimony he provides (SB 15) establishes that Canich was authorized to make a request to Dr. Floyd for an elemental analysis of the product Canich made. Neither Dr. Burkhardt nor Dr. Floyd have testified that elemental analysis was either necessary or required.

Stevens (SB 16-23) further directs our attention to the testimonies of Dr. Bercaw, an Exxon consultant (SB 16), Dr. Cynthia Day, the president of Crystalytics Company and the person who performed the x-ray crystallographic analyzes mentioned earlier (SB 18), Dr. Jordan, a Professor of Chemistry at the University of Iowa, and Dr. Parkin, a Professor of Chemistry at Columbia University. Stevens (SB 23-27) also mentions Dr. Stevens, Dr. Wilson, and other Stevens' experts (i.e., Drs. Marks, Ibers, Landis and Waymouth) and cities publications. In each instance, Stevens seeks to make the case that elemental analysis is the routine, simple, well-established and widely accepted means of identifying a new compound. Stevens also cites Berges v. Gottstein, 618 F.2d 771, 772-73, 205 USPQ 691, 692-93²⁶ (CCPA 1980), one of the facts in the case being that an independent laboratory conducted an elemental analysis to confirm the identity of a compound. According to Stevens, "[e]ven the legal authorities upon which Canich relies endorse the importance of elemental analysis" (SB 25-26).

There is no dispute that elemental analysis is a routine, simple, well-established and

"standard elemental analysis determining carbon, hydrogen, and nitrogen percentages."

widely accepted means of identifying a new compound. Furthermore, Canich does not appear to disagree that elemental analysis was a standard procedure used in Exxon. However, it is not clear why Canich should be limited to that procedure. The question of whether Canich recognized the compound it made was AZ does not turn on whether any specific procedure is the routine, simple, well-established and widely accepted method or the standard used in Exxon. Rather it depends on whether the analytical test used to perform the analysis shows the result to be the compound Canich alleged it to be.²⁷ In our view, that is what Canich has done.

We are not persuaded by Stevens' argument that Canich needed to conduct routine analytical testing necessary to establish what was synthesized. Whether or not Canich recognized or appreciated that it prepared AZ depends not on whether Canich conducted an elemental analysis, as conclusive as such a test might be, but on the evidence as a whole. That evidence, as we have discussed supra, shows more likely than not that Canich recognized and appreciated that it had prepared AZ prior to the August 31, 1989 priority date. Accordingly, Stevens has not persuasively shown that Canich did not recognize or appreciate that it prepared AZ.

(2) NMR

According to Stevens (SB 27), "Canich's proton NMR spectra could not have been

Compare with the standard used in evaluating tests establishing utility: "[T]he inquiry is not what kind of test was conducted, but whether the test conducted showed that the invention would work as intended in its contemplated use." Eastern Rotorcraft Corp. v. United States, 384 F.2d 429, 431, 155 USPQ 729, 730 (Ct. Cl. 1967).

used to reasonably identify the new metal complexes Canich alleges were synthesized." In other words, Stevens is arguing that, irrespective of the fact that Canich did not perform an elemental analysis, the NMR spectra provided by Canich does not show the result to be the compound Canich alleges it to be.

Stevens (SB 27-36) discusses a number of problems with respect to Canich's NMR spectra. This includes operator errors (SB 27), that the spectra provide information only about protons (SB 27), the poor quality of the spectrum due to, for example, impurities (SB 28), lack of proper referencing of the spectrum (SB 28-30), improper labeling and maintaining of the spectra (SB 30), potential pulse delay problems (SB 30-31), poor shimming of the spectra resulting in, for example, poor peak resolution (SB 31), and poor technique as evidenced by different chemical shifts for the same compound tested in the same solvent (SB 32-33). As a result, according to Stevens, "Canich's proton NMR spectra ... are consistent with the presence of unintended bis-Cp metal complexes that were the likely result of Canich's syntheses" (SB 33). Stevens (SB 33-37) goes on to discuss the similarity one can expect in proton NMR spectra, especially in terms of chemical shifts, for both unintended and the intended compounds.

The evidence does not establish, and Stevens does not dispute it, that Canich's NMR spectra are inconsistent with an identification of AZ. Stevens argues only that Canich's proton NMR spectra are consistent with the preparation of other unintended bis-Cp metal complexes. However, even if that were the case, Canich need not and does not

rely solely on the NMR spectra for the analytical testing necessary to establish that it recognized and appreciated that it prepared AZ.

Consistent with Young v. Bullitt, 233 F.2d 347, 352, 110 USPQ 55, 59²⁸ (CCPA 1956), Canich (CB 48-49) submits that the evidence, taken as a whole, establishes "by a preponderance of the evidence that Dr. Canich actually prepared the mono-Cp compounds that she thought she synthesized." Canich (CB 55) points out that, in addition to the proton NMR, X-ray crystallography was, for example²⁹, also performed. In this regard, Canich (CRB 50) argues that Dr. Day's X-ray crystal structure determination of a sample of AZ "is consistent with the X-ray crystal structure data of compound AZ recently published by Dr. Petersen."

Stevens (SB 38-39) responds that (1) the sample Dr. Day characterized is not related to any of Dr. Canich's synthesis experiments and (2) the crystallographic analysis Dr. Day performed could not detect the difference between, for example, a niobium and a zirconium complex. However, Stevens does not dispute that, on February 28, 1989, well before the August 31, 1989 priority date, Dr. Canich sent a letter with a sample alleged to be of AZ to Dr. Day for x-ray crystallographic characterization (CB Tab 3, p. 3). Dr. Day

²⁸ "[I]t is not necessary that the product relied on by a party ... be identified with absolute certainty, or that a complete analysis should invariably be made. It is sufficient if the evidence, taken as a whole, establishes, with reasonable certainty, the identity of the product."

²⁹ Other analytical testing that Canich argues establishes that Canich recognized AZ when it was made are shown by "subsequent work by others (including Stevens) showing that the NMR spectra Dr. Canich obtained are in good agreement with the preparation of compound AZ by others" (CB Tab 3, p. 2) and Dr. Turner's derivatization work involving proton and carbon-13 NMR of the dimethylated AZ compound. CB Tab 3, pp. 9-12.

began the determination on March 13, 1989 (CB 18) and telephoned Dr. Canich on March 14, 1989, with a final report on August 9, 1989, establishing that the sample had a structure consistent with AZ (CB Tab 3, p. 9). This too Stevens does not dispute. Given that AZ was prepared four times, the last time in October 1988, and that it was the subject of continuing analyses, through NMR and other techniques, the preponderance of the evidence is that around March 1989 Dr. Day tested a sample that had been earlier synthesized by Dr. Canich. The preponderance of the evidence also supports a conclusion that the sample Dr. Day characterized was a zirconium rather than a niobium complex. The x-ray data Dr. Day provided to Dr. Canich is consistent with later x-ray data provided by Dr. Petersen for the zirconium complex (CB 39). Also, as we noted supra, Canich (CRB 48) "was not starting from scratch." Canich sought to prepare a zirconium alternative to the scandium compounds previously prepared by Dr. Bercaw.

Given the evidence taken as a whole, the preponderance of the evidence shows that Canich confirmed through analytical testing, including proton NMR spectra and x-ray crystallographic techniques, that the product it had obtained was the product Canich identified as AZ.

B) Record Keeping

Stevens also argues that Canich's record keeping was such that there is little or no evidence to substantiate Canich's claim that it in fact made a compound within the scope of

the count.

The issue of documentation is raised by Stevens with respect to:

- laboratory practices as to preparation (SB 40; 44-45; 48-49);
- analytical data (e.g., proton NMR spectra) (SB 41; 42-44; 46-47; 52-59);
- utility (i.e., polymerization) (SB 42; 60);
- corroboration as to preparation (SB 41; 47; 50); and,
- corroboration as to utility (SB 51-52).

At issue here is whether Canich has established with a preponderance of the evidence that it recognized or appreciated AZ when it was prepared. Accordingly, we limit our discussion here to the documentation as to laboratory practices and analytical data and discuss the documentation issue with respect to utility and corroboration infra in the sections entitled "Utility" and "Corroboration", respectively.

According to Stevens, Stevens takes the position that "Canich's synthesis experiments are not properly documented" (SB 48). The reason for this is, according to Stevens, that "Dr. Canich failed to follow Exxon's routine practices and procedures for properly documenting her research efforts" (SB 39-60). Stevens argues that Canich violated Exxon procedures on how to record analytical data (SB 42). Certain passages referred to as "Exxon's protocol" are reproduced in Stevens' Brief (SB 41-45), wherein Stevens compares Canich's actions to the protocol, concluding therefrom that Canich did not exercise the necessary care to ensure that no question of reliability and authenticity could be raised (SB 44). According to Stevens (SB 45),

Canich's failures to have the experiments properly dated, signed and witnessed has necessitated Canich's attempt to invoke the so-called

"cohesive web" theory. However, these same failures, as well as evidence of alteration of data, preclude reasonable reliance on Canich's laboratory notebooks or the analytical results alleged to relate thereto as evidence of priority of invention or discovery.

Canich responds (CRB 22-36) with a discussion of the documentation on record.

According to Canich, the documentation on record shows that Dr. Canich's practices conformed with Exxon's policy concerning record keeping practices.

The parties disagree as to whether Canich followed standard record keeping practices set forth by Exxon for its researchers. However, the issue here is whether Canich has shown, through contemporaneous records, that it actually prepared, for example, AZ and recognized it before the August 31, 1989 priority date. The issue is not whether Canich violated the rules set forth by Exxon, even though the rules may have been for the purpose of clarifying questions of priority.

In our view, the record is extensive. The record (see e.g., CR Appendix) is replete with documents showing in clear and detailed language the steps Canich followed in actually preparing and analytical testing mono-Cp compounds. Based on a preponderance of the evidence, we find that Canich properly documented the results to such an extent that, more likely than not, Canich recognized and appreciated that it prepared a compound within the scope of the count.

Utility

Whether a practical utility has been established for a novel compound is a question of fact. To show reduction to practice, the junior party must demonstrate that the invention is "suitable for its intended purpose." Steinberg v. Seitz, 517 F.2d 1359, 1363, 186 USPQ 209, 212 (CCPA 1975).

Canich argues that "each mono-Cp compound was tested for utility as a component of a polymerization catalyst" (CB 20). According to Canich (CB 20-21), polymerization experiments were conducted and the resulting polymers were tested by gel permeation chromatography. The compounds were active catalysts for olefin polymerization and, according to Canich (CB 21), "typically produced narrow molecular weight distributions."

Canich's Brief (CB 26-29; Tab 2, pp. 12-16) provides a detailed description of a polymerization run with AZ conducted by Mr. Upton on September 1, 1988. Documentation for this run is alleged to be provided by a laboratory notebook and a preprinted sheet taped into the notebook (CB 26). According to Canich (CB Tab 2, p. 14), Mr. Upton's run is corroborated by Dr. Canich's "September Update" to Dr. Chow. Other polymerization runs are also discussed. (e.g., at CB 29-30). This shows that Canich recognized a specific practical utility for an embodiment of the count.

In rebutting Canich's evidence of utility, Stevens argues that "Canich's proofs strongly suggest that bis-Cp metal complexes were responsible for the observed polymerization activity" (SB 101) and that Canich "failed to take any steps to identify ... the

components in the catalyst system that were responsible for the polymerization that she reported" (SB 102). Steven's rebuttal is unpersuasive.

There is no dispute that Canich used a compound it had synthesized to conduct the polymerization. Moreover, Stevens provides no evidence that the polymerization activity that occurred when Canich conducted its experiments could not have been obtained from the use of a mono-CP compound. Together with the preponderance of the evidence showing that Canich actually prepared AZ and recognized and appreciated that it had made AZ, the circumstantial evidence weighs in favor of a conclusion that, when Canich conducted the polymerization, it did so with a mono-Cp compound.

Accordingly, the preponderance of the evidence establishes that the mono-Cp compound Canich prepared operated and was suitable for its intended polymerization purpose .

Corroboration

Canich "must provide independent corroborating evidence in addition to his own statements and documents." Hahn v. Wong, 892 F.2d 1028, 1032-33, 13 USPQ2d 1313, 1317 (Fed. Cir. 1989). Inventor's testimony as to the inventor's activities in reducing an embodiment of the count to practice must be corroborated by some independent evidence. Reese v. Hurst, 661 F.2d 1222, 1226, 211 USPQ2d 936, 940 (CCPA 1981).

Canich (CB 55) argues that it has "provided a cohesive web of corroborative

evidence." Canich's case for corroboration is presented in its brief at CB 53-56, supported by facts discussed at CB 5-21. Stevens presents a rebuttal to Canich's position, in part, at SB 103-109. Canich replies to Steven's rebuttal at CRB 65-92.

We turn now to the evidence and determine whether it is sufficient to show that Canich has established a "cohesiveness in the web of allegedly corroborating evidence," Berges v. Gottstein, 618 F.2d 771, 776, 205 USPQ 691, 695³⁰ (CCPA 1980). In doing so, we apply a "rule of reason," Anderson v. Pieper, 442 F.2d 982, 985, 169 USPQ 788, 790 (CCPA 1971). We remain mindful that "[c]orroboration is not a ritual but a method for determining the veracity of the testimony," Mattor v. Coolegem, 530 F.2d 1391, 1395, 189 USPQ 201, 203 (CCPA 1976).

Actual Preparation

According to Canich, corroboration for the actual preparation of a mono-Cp

³⁰ "A different situation existed in Mikus v. Wachtel, *supra*, [Mikus v. Wachtel, 542 F.2d 1157, 191 USPQ 571 (CCPA 1976)] cited by the Board as requiring acts entirely independent of the inventor. At first glance, the problem there appears similar to the instant one. An unwitnessed notebook was submitted with no firsthand knowledge of a synthesis by co-workers. Independent analyses of a compound within the count, however, were performed. A critical difference in Mikus was the lack of cohesiveness in the web of allegedly corroborative evidence [our emphasis] on which Mikus relied. As this court noted, consistency among the individual components of evidence was not clearly established. For example, some of the analyses were performed on compounds not carrying the proper label. Significantly, the inventor had specifically assigned a suffix to his label which denoted the completion of a necessary process step in the synthesis of the composition of the count. Such a situation does not exist in this case."

Nor can we agree with the board's statements implying that corroborators must have been present at the actual work site or have known something about it other than what was reported to them, for which it cited Patterson v. Clements, 30 CCPA 1262, 136 F.2d 1002, 58 USPQ 539 (1943), a case involving a single corroborating witness. Corroborative testimony does not necessarily have to be an actual witnessing of the reduction to practice by one who understands what is going on in order to be adequate. Sufficient circumstantial evidence of an independent nature can satisfy the corroboration rule."

compound within the scope of the count, such as AZ, is established by, for example, the following:

- Dr. Chow (CR 137) declares that Dr. Canich joined his Polymer Research Group at Exxon in September 1987;
- Dr. Chow (CR 137) asked Dr. Canich to prepare a research proposal along the lines of the groups areas of interest - i.e., the "EX-300 project" involving group 4 metal bis-Cp compounds - and he (along with Drs. Burkhardt, Hlatky, Turner and Welborn) received such a proposal (CX 1338 and 75) on or about October 23, 1987;
- Dr. Chow (CR 138-139), among others, decided that Dr. Canich begin work on one of her proposed research projects, to work on group 5 metal compounds, but to spend half her time on the EX-300 catalyst system;
- Dr. Chow (CR 140), among others, received a Monthly Update on or about May 2, 1988, from Dr. Canich with respect to her work on the EX-300 project wherein she states "Zirconium chemistry was initiated ... [t]he plan of attack ... will most likely include ... mono-Cp zirconium complexes ... which may or may not be bridged to the Cp ring" (CX 1285);
- Dr. Chow (CR 141) declares that Dr. Canich, also around May 2, 1988, "also reported about a scandium containing compound prepared by Bercaw et al. that reportedly polymerizes propylene";
- a copy of a purchase requisition form (CX 81) prepared by Dr. Canich and approved by Dr. Chow (signed April 21, 1988) shows certain chemicals to be purchased from Aldrich which Dr. Chow (CR 142) declares "were to be used to prepare the zirconium $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-Bu})\text{ZrCl}_2$ ";
- a meeting (summarized in Dr. Chow's May 17, 1988 memorandum - CX 1353) was held on May 6, 1988 and, according to Dr. Chow's Declaration (CR 142), attended by Drs. Canich, Chow, Burkhardt, Hlatky, Pannell, Turner and Welborn and Mr. Finkelstein, in which Dr. Canich's work on mono-Cp Group 4 compounds was discussed;
- on May 19, 1988, Dr. Hlatky (CR 223, CX 1286) signed a patent memorandum "listing Dr. Canich as the inventor of 'Mono-Cp Group 4 complexes with MAO [methylalumoxane]: Olefin Polymerization Catalysts'";
- the chronology of events set forth above is also corroborated by Dr. Turner (CR 544-557);
- Dr. Chow (CR 148) prepared a weekly update on June 6, 1988 and provided a copy to his superior, Dr. Matagna, wherein it states that "Dr. Canich ('Joann' [sic]) 'is working on the synthesis of the amide bridged zirconium metallocene' and '[t]his is the first mono Cp DICOP that we are attempting to synthesize and it will be used in the EX-300 system as well'"

- Dr. Turner (CR 547) declares that, in the summer of 1988, he became aware that "Dr. Canich was lacking the tetra methyl-cyclopentadienyl compound for use in her experiments for synthesizing mono-Cp compounds", whereupon Dr. Turner
 - called Dr. Bercaw for a preparation of the compound, which was sent by Pamela Shapiro (CX 91);
 - prepared, along with Dr. Welborn and T.A. Mathew, the compound from the preparation sent by Shapiro; and,
 - "gave my portion of the yield to Dr. Canich for her use in preparing the mono-Cp compounds" (CR 548-549);
- laboratory notebooks of Dr. Canich were witnessed and signed by Drs. Welborn, Turner or Hlatky (CX 1373, CX 1400) and monthly updates for June, July and August, 1988, were distributed (CX 1332, 1339, 1340), all indicating Dr. Canich's continued work on and final preparation of the mono-Cp compound; and,
- Dr. Turner (CR 550) declares that, by September 30, 1988, he obtained a quantity of the $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-Bu})\text{ZrCl}_2$ finally synthesized by Dr. Canich and gave this to Mr. Zamora who, at Dr. Turner's request, performed a dimethylation experiment on the sample (CX 1314).

Recognition or Appreciation

According to Canich, corroboration for the recognition or appreciation of the synthesized mono-Cp compound is established by, for example, the following:

- On March 13, 1989, Dr. Day completed an x-ray crystallographic analysis of a sample received from Dr. Canich and Dr. Day reported to Dr. Canich (CX 188) that the sample was $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-Bu})\text{ZrCl}_2$; and,
- Dr. Turner (CR 550-554) declares that, by way of Mr. Zamora's work, he characterized the dimethylated derivative (i.e., $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-Bu})\text{ZrMe}_2$) of Dr. Canich's compound with proton NMR, and with the assistance of Dr. Eckman did the same with carbon-13 NMR, and thereby verified that Dr. Canich supplied the compound $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-Bu})\text{ZrCl}_2$.

Utility

According to Canich, corroboration for the utility of the synthesized mono-Cp compound is established by, for example, the following:

- Mr. Upton (CR 601-613) declares that he performed separate polymerization

experiments "for Dr. Canich using various mono-Cp compounds prepared and supplied to me by Dr. Canich and using MAO as the cocatalyst during the period from December 1988 through May of 1989" (CR 601).

The corroborating evidence discussed supra do not appear to be in dispute. Stevens argues (SB 105-109) instead that

- "Dr. Canich clearly did not follow the practices and procedures required by Exxon to document and confirm that a metal complex of the count was made." SB 104;
- "Canich did not perform the routine tests necessary to establish the identity or whatever was made." SB 106;
- "Canich also did not follow Exxon's routine practices for making, handling and documenting her research efforts." SB 107;
- "... the credibility of Canich's notebooks, proton NMR and carbon NMR spectra is hopelessly compromised." SB 108
- "It is also telling that Canich fails to offer any analysis of a retained sample of the compounds that she made, with no explanation as to why no sample of the many that were claimed to have been made has surfaced."

Stevens' arguments against Canich's corroborating evidence are unpersuasive.

These arguments go less to the issue of corroboration than to Canich's position with respect to establishing each element of the two-prong test for demonstrating an actual reduction to practice. We have addressed similar arguments in our discussion supra of the two-prong test. Suffice it to say that the issue here is whether Canich has established independent corroborating evidence. Whether or not Dr. Canich followed, for example, certain previously prescribed procedures is irrelevant to the issue of corroboration.

In our view, we do not see that Stevens fairly and reasonably attacks the issue of corroboration. The "rule of reason" involves a reasoned determination as to the credibility

of the inventor's story based on an examination, analysis and evaluation of the record as a whole. Berges v. Gottstein, 618 F.2d 771, 776, 205 USPQ 691, 695 (CCPA 1980). To that end, there is a cohesiveness in the web of allegedly corroborating evidence. Canich provides independent evidence through declarations of researchers other than Dr. Canich, and with supporting documents, to show a chronology of events beginning in September of 1987, when Dr. Canich began work with Dr. Chow, through the conception phase during which a research proposal was submitted with a corresponding meeting, purchase of ingredients in April 1988, preparation of an intermediate in the summer of 1988, and finally in August 1988, with the preparation of an embodiment within the count. Similarly, independent evidence has been provided showing that, prior to the priority date of August 31, 1989, Dr. Canich recognized and appreciated what she made and established a practical utility for the synthesized compound. Accordingly, we are satisfied that, based on the record as a whole, the preponderance of the evidence weighs in favor of a conclusion that independent corroborative evidence exists for Dr. Canich's testimony in support of Canich's position that an actual reduction to practice occurred prior to the August 31, 1989 priority date. Canich has, in our view, satisfactorily shown the veracity of Dr. Canich's testimony.

For the foregoing reasons, we find that, more probable than not, prior to August 31, 1989, Canich actually prepared AZ, recognized that the compound it prepared was AZ, and recognized a specific practical utility for it. Canich's testimony in support thereof is

sufficiently corroborated. Accordingly, Canich has satisfied its burden and prevails as to the issue of priority.

OTHER ISSUES

I. Stevens includes a section in its brief entitled "**XII. Stevens' Claims Are Patentable**" (SB 169-187). Within this section, two requests are made.

A. Stevens (SB 170) "urges the Board to affirm Judge Downey's decision [in the decision on motions (Paper No. 299)] allowing the motion [contingent belated motion 26] pursuant to 37 CFR §1.633(c)(2) (Paper No. 122)" to add claims 111-118 to the interference as corresponding to the count. The request is made on the grounds (SB 170) that "there is no real dispute that those claims are patentable and Canich's objection to the motion adding those claims is without merit." This request is dismissed as MOOT because claims 111-118 have already been added as corresponding to the count as part of the Redeclaration Notice (see footnote 7).³¹

B. Stevens (SB 172) states

Judge Downey correctly granted Stevens' motion to add new claims 111-118. The Board should affirm that decision. In that event, Stevens asks that all earlier claims [1, 2, 22, 49-56, 102, 103, and 109; see SB 170] be canceled, so that the Board need not consider Canich's lengthy and confusing unpatentability argument with respect to Stevens' first set of amended claims [49-56, 102, 103, and 109; see

We also point out that a party cannot remove involved claims from an interference and thus avoid their cancellation under 35 U.S.C. 135(a) in the event of a final judgment adverse to that party. See Nelson v. Drabek, 212 USPQ 98, 99 (Comm'r Pats. 1979) and Theeuwes v. Bogentoft, 2 USPQ2d 1378, 1379 (Comm'r Pats. 1986).

SB 171]. Those arguments have no bearing on Stevens' second set of amended claims [111-118].

As we understand it, Stevens is requesting that claims 1, 2, 22, 49-54, 102, 103 and 109, all of which except claim 22 are designated to correspond to the count, be cancelled.

Stevens makes this request "so that the Board need not consider Canich's ... unpatentability argument with respect to" claims 49-56, 102, 103, and 109. However, that section in Canich's Brief (CB 57-135) which is devoted to the unpatentability of Stevens' claims has been redacted in accordance with an agreement of the parties. Therefore, Stevens provides us with no good reason to grant the request. Moreover, we see no reason to make an additional determination with respect to cancelling 1, 2, 22, 49-56, 102, 103, and 109 given that Stevens has not prevailed on the issue of priority and is therefore nevertheless not entitled to claims 1, 2, 49-54, 102, 103, 109 and 111-118 of their application corresponding to Count 2. Accordingly, Stevens' request to cancel claims 1, 2, 22, 49-56, 102, 103, and 109 is DENIED.

.II. Stevens includes a section in its brief entitled "**XIII. Metal Complexes in the +3 Oxidation State Are Patentably Distinct**" (SB 187-227). In it, Stevens states:

The preliminary decision granting Stevens' Motion Nos. 4 and 37 is no longer challenged by Canich. In the Preliminary Decision, the APJ ruled that metal complexes in the +3 oxidation state are patentably distinct from the count. See Decision on Preliminary Motions, Paper No. 299, at pages 19-21 and 26. Pursuant to the parties' agreement, Steven's Motion Nos. 4 and 37 are no longer opposed and Canich's Motion Nos. 23 and 26 have been withdrawn.

Since no action is being requested, no further comment is needed.

III. Stevens includes a section in its brief entitled "**XIV. The Titanium Subgenus Is A Separately Patentable Invention**" (SB 228-240). Therein Stevens (SB 230) states that

The record unquestionably shows that the titanium subgenus is separately patentable from zirconium, hafnium, titanium genus, and that Stevens is the inventor of the titanium subgenus. The Board's Decision should clearly reflect these facts in order to resolve all issues between the parties.

Consequently, the Board should reverse the decision denying Stevens' Motion No. 12 to add the titanium subgenus count. ...

As Stevens (SB 229) acknowledges, the titanium subgenus was the subject matter of the count in a separate interference - 103,819 - declared between Canich's U.S. Patent 5,096,867 and application 08/872,119 and Stevens' 07/545,403 application. Accordingly, a determination has already been made that the titanium subgenus is separately patentable from the zirconium, hafnium, titanium genus involved in this interference. Judgment in interference 103,819, as to the subject matter of the count, i.e., the titanium subgenus, was awarded to Stevens and Stevens was entitled to claims in its 07/545,403 application corresponding to the count. Accordingly, the request to review the APJ's decision in the Decision on Motions denying Stevens' Motion No. 12 to add a titanium subgenus to this interference is MOOT.

IV. Ganich (CB 180-214) provides a section entitled "**Canich's Claims.**" In this section, Canich (CB 181) urges that "the Canich claims that should be designated to new Count 2, and should be in Canich's '428 application are Claims 4, 5, 35-41, 44 and 45." The claims that are presently designated to correspond to Count 2 are Claims 2,

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4-6, 25-26, 35-41, 44 and 45. Canich is seeking to cancel Claims 2, 6, 25 and 26 which have been designated to correspond to the count "in order to either remove unabridged mono-Cp subject matter or to moot arguments made by Stevens." CB 181. Furthermore, Canich "reargues the appropriateness of canceling and amending claims to the '428 application." CB 182.

As we understand it, in effect, we are being requested to review Canich Preliminary Motion 17 since that is the Canich motion that seeks to cancel claims 2, 6, 25 and 26 and amend certain claims (i.e., claims 4 and 5, see CB 198, 202-206).

In Preliminary Motion 17 (Paper No. 170), Canich moved pursuant to 37 CFR §§ 1.633(i) and 1.633(c)(2) to redefine the interfering subject matter by cancelling claims 2, 6, 25, 26 and 28 and replacing them with new claims 30-45 to Canich's '428 application, as well as rewriting claims 4 and 5 in independent form. Preliminary Motion 17 is contingent on Canich Preliminary Motion 16 (Paper No. 169) wherein Canich moves pursuant to 37 CFR §§ 1.635 and 1.645(b) for an order permitting the late filing of, among others, Canich Motion 17.

In the Decision on Motions (Paper No. 299, p. 15), the APJ granted the motion to add claims 35-41 and 44-45. Accordingly those claims have been designated to correspond to the count and no further action with respect to those claims is needed. As to claims 30-34, 42 and 43, the Decision on Motions notes (Paper No. 299, p. 15, footnote 5) that "Canich withdrew her request to add claims 30-34, 42 and 43."

Accordingly, we take no position as to claims 30-34, 42 and 43. With respect to claim 28, the Decision on Motions (p. 11) dismissed the Canich motion (Canich Preliminary Motion 5) to add claim 28 and therefore claim 28 has not been designated as corresponding to the count. Accordingly, that aspect of Canich Motion 17 that moves to cancel claim 28 has been rendered moot.

With respect to the remaining aspects of Canich motion 17, i.e., cancelling claims 2, 6, 25 and 26 and amending claims 4 and 5 in independent form, we note that the parties have agreed (paper no. 557) that Canich Motion 17, as well as Canich Motion 16, are pending without opposition. Accordingly, Canich Motion 16 is GRANTED and the belatedness of Canich Motion 17 is excused. Canich Motion 17 is GRANTED to the extent that Canich can cancel claims 2, 6, 25 and 26 before the examiner upon termination of this interference and during subsequent continuation of ex parte prosecution of the Canich '428 application. With respect to that part of Canich Motion 17 that moves to amend claims 4 and 5 in independent form, the motion is GRANTED subject to a determination by the examiner of the appropriateness of the claim language and the examiner's consideration of issues of patentability. Upon termination of this interference and during subsequent continuation of ex parte prosecution of the Canich '428 application, the examiner should review amendments to claims 4 and 5 and determine their patentability not inconsistent with the decision in this interference.

JUDGMENT

For the foregoing reasons, judgment as to the subject matter of the count is entered in favor of junior party, Jo Ann M. Canich and judgment is awarded against senior party James C. Stevens, Francis J. Timmers, David R. Wilson, Gregory F. Schmidt, Peter N. Nickias, Robert K. Rosen, Gregory W. Knight and Shih-Yaw Lai.

Having decided all the issues properly raised by the parties in their briefs, we now enter judgment in this interference pursuant to our authority under 37 CFR § 1.658(a). Accordingly: On this record, we hold the following:

Jo Ann M. Canich, the junior party, is entitled to a patent containing claims 2, 4-6, 25, 26, 35-41, 44 and 45 of their application corresponding to Count 2.

James C. Stevens, Francis J. Timmers, David R. Wilson, Gregory F. Schmidt,
Peter N. Nickias, Robert K. Rosen, Gregory W. Knight and Shih-Yaw Lai, the senior party,
are not entitled to a patent containing claims 1, 2, 49-54, 102, 103, 109 and 111-118 of
their application corresponding to Count 2.

Marc L. Carr

MARC L. CAROFF //
Administrative Patent Judge

Mary F. Downey
MARY F. DOWNEY
Administrative Patent Judge


HUBERT C. LORIN
Administrative Patent Judge

) BOARD OF PATENT
)) APPEALS
)) AND
)) INTERFERENCES

Interference No. 102,953

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The opinion in support of the decision being entered today was not
written for publication and is not binding precedent of the Board.

Paper No. 681

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE BOARD OF PATENT APPEALS
AND INTERFERENCES

MAILED

JO ANN M. CANICH
Junior Party¹,

SEP 28 2001

PAT. & T.M. OFFICE
BOARD OF PATENT APPEALS
AND INTERFERENCES

v.

JAMES C. STEVENS, FRANCIS J. TIMMERS,
DAVID R. WILSON, GREGORY F. SCHMIDT,
PETER N. NICKIAS, ROBERT K. ROSEN,
GEORGE W. KNIGHT and SHIH-YAW LAI
Senior Party².

Patent Interference No. 102,954³

R E C E I V E D

OCT - 2 2001

FINAL DECISION - On Brief⁴

FISH & NEAVE - PATENT DEPT.
REFERRED TO _____
NOTED BY _____

Before CAROFF, METZ, and LORIN, Administrative Patent Judges.

LORIN, Administrative Patent Judge.

¹ U.S. Patent 5,026,798 based on Application 07/581,817, filed on September 13, 1990 and issued June 25, 1991; Application 07/973,107 for reissue of U.S. Patent 5,026,798, filed November 6, 1992; and, Application 07/676,690, filed March 28, 1991. Assigned to Exxon Chemical Patents, Inc., a company wholly owned by Exxon Corporation.

² Application 07/545,403, filed July 3, 1990. Assigned to The Dow Chemical Company.

³ This interference is related to interference 102,953 on which a Final Decision was mailed on December 29, 2000.

⁴ Final Hearing was waived by the parties.

Interference No. 102,954

This is a Final Decision in the interference proceeding involving Canich U.S. Patent 5,026,798, filed on September 13, 1990, Reissue Application 07/973,107, filed November 6, 1992, and Application 07/676,690, filed March 28, 1991; and, Stevens et al. (Stevens) Application 07/545,403, filed July 3, 1990.

Stevens has been accorded the benefit of an earlier filing date.⁵ Accordingly, Stevens is designated the senior party in this interference. 37 CFR §§ 1.657 and 1.601(m).

Count 2⁶, the sole count at issue, defines the interfering subject matter.⁷

⁵ Canich's '798 patent has been accorded the benefit of the September 13, 1989 and June 4, 1990 filing dates of earlier Canich applications 07/406,945 (now abandoned) and 07/533,245 (now U.S. Patent 5,055,438, issued October 8, 1991), respectively.

Canich's '107 reissue application has been accorded the benefit of the September 13, 1989 and June 4, 1990 filing dates of earlier Canich applications 07/406,945 (now abandoned) and 07/533,245 (now U.S. Patent 5,055,438, issued October 8, 1991), respectively.

Canich's '690 application has been accorded the benefit of the September 13, 1989, June 4, 1990, and September 13, 1990 filing dates of earlier Canich applications 07/406,945 (now abandoned), 07/533,245 (now U.S. Patent 5,055,438, issued October 8, 1991), and 07/581,817 (now U.S. Patent 5,026,798, issued June 25, 1991), respectively.

Stevens' '403 application has been accorded the benefit of the October 27, 1989 and August 31, 1989 filing dates of earlier Stevens applications 07/428,283 and 07/401,3454, respectively.

See the Redeclaration of the interference (Paper No. 239); made pursuant to a decision on preliminary motions (Paper No. 237).

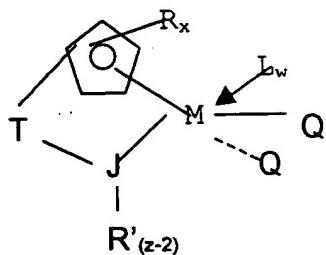
⁶ The Redeclaration Notice (Paper No. 239, p. 1) deleted original Count 1 and substituted Count 2.

⁷ 37 CFR §1.601(f).

Count 2

A process for polymerizing one or more olefins, diolefins or acetylenically unsaturated compounds comprising the steps of

- (i) contacting an olefin, diolefin or acetylenically unsaturated monomer at a temperature and pressure sufficient to polymerize such monomer with a catalyst system comprising;
- (A) an alumoxane, and
 - (B) a group IV-B transition metal component of the formula



where M is Zr, Hf or Ti in its highest formal oxidation state;

R is a substituent group with "x" denoting the degree of substitution (x = 0, 1, 2, 3 or 4) and each R is, independently, a radical selected from a group consisting of C₁-C₂₀ hydrocarbyl radicals, substituted C₁-C₂₀ hydrocarbyl radicals substituted C₁-C₂₀ hydrocarbyl radicals wherein one or more hydrogen atoms is replaced by a halogen radical, an amido radical, a phosphido radical, an alkoxy radical or any other radical containing a Lewis acidic or basic functionality, C₁-C₂₀ hydrocarbyl-substituted metalloid radicals wherein the metalloid is selected from the Group IV-A of the Periodic Table of Elements, and halogen radicals, amido radicals, phosphido radicals; alkoxy radicals, alkylborido radicals or a radical containing Lewis acidic or basic functionality, or at least two adjacent R-groups are joined forming C₄-C₂₀ ring to give a saturated or unsaturated polycyclic cyclopentadienyl ligand;

(JR'_{z-2}) is a heteroatom ligand in which J is an element with a coordination number of three from Group V-A or an element with a coordination number of two from Group VI-A of the Periodic Table of Elements, and R' is a radical selected from a group consisting of C₁-C₂₀ hydrocarbyl radicals, substituted C₁-C₂₀ hydrocarbyl radicals where one or more hydrogen atoms is replaced by a halogen radical, an amido radical, a phosphido radical, an alkoxy radical or a radical containing a Lewis acidic or basic functionality, and "z" is the coordination number of the element J;

each Q is, independently, any univalent anionic ligand or two Q's are a divalent anionic chelating ligand, provided that Q is different from



T is a covalent bridging group containing a Group IV A or V A elements; L is a neutral Lewis base where "w" denotes a number from 0 to 3; and
(ii) recovering a polymer.

The parties' claims which correspond to Count 2 are:⁸

- Canich's '690 application: claims 18-33;
- Canich's patent: claims 1-13;
- Canich's reissue: claims 1-13; and,
- Stevens' '403 application: claims 7-10, 14/10, 29-32.

The parties filed the following Briefs and Reply Briefs:

CB⁹ Canich Brief (redacted), filed November 8, 1999 (Paper No. 564)

SB Stevens Brief (redacted), filed December 10, 1999 (Paper No. 567)

CRB Canich Reply Brief (redacted), filed February 4, 2000 (Paper No. 569).

Canich filed a Record and Exhibits (Paper No. 479 and 480, respectively).¹⁰

Stevens filed a Record and Exhibits (Paper Nos. 457 and 458, respectively).¹¹

⁸ See the Redclaration Notice (Paper No. 239, p. 7).

⁹ Hereinafter, the Briefs and Reply Briefs will be designated by these abbreviations followed by page number.

¹⁰ Hereinafter designated as CR and CX followed by page and exhibit number, respectively.

¹¹ Hereinafter designated as SR and SX followed by page and exhibit number, respectively.

The issues presented for our decision in the parties' briefs include Canich's priority case and other issues¹². An issue of no interference-in-fact has been raised and will be addressed first. We will then address the priority issue, followed by a discussion of other issues raised by the parties in their briefs.¹³

INTERFERENCE-IN-FACT

Canich's Brief (CB 58-76) provides for a section entitled "**There Is No Interference-In-Fact Between The Canich '798 Patent (And Its Reissue Application) And The Stevens '403 Application**" wherein Canich argues that "[t]he decision denying Canich Motion No. 1 was manifestly erroneous. Canich Motion No. 1 should have been granted." CB 60. Canich Motion 1 (paper no. 23) was denied by the

¹² The parties have provided the following Statements of the Issues:

Canich's Statement of the Issues (CB xviii)

1. Has Canich demonstrated that it is entitled to priority on Count 2 by a preponderance of the evidence based on actual reductions to practice prior to Stevens' earliest filing date of August 31, 1989?

2. Is there an interference-in-fact between Canich's '798 patent and reissue '107 application and Stevens' '403 application?

Stevens' Statement of the Issues (SB xli-xlii)

1. Has Canich proven by credible evidence that she actually used a process of the count prior to Stevens' effective filing date?
2. Has Canich proven by credible evidence that she performed sufficient testing to recognize the process of the count and attribute the observed polymerization to the catalyst system recited by the count?
9. Should the Board permit Stevens to add claims 129-136 which moot many of Canich's unpatentability arguments?
10. Are Stevens claims 129-136 patentable?
12. Do Stevens' claims that require no-alumoxane cocatalysts define a separate patentable invention?
13. Is the titanium subgenus patentably distinct?

¹³ Matters not raised in a parties' brief are ordinarily regarded as abandoned. Photis v. Lunkenheimer, 225 USPQ 948 (Bd. Pat. Int. 1984).

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APJ in the Decision on Motions (paper no. 237, pp. 7-8). Accordingly, as we understand it, Canich is requesting that the Board review the APJ's decision denying the motion. The burden is on Canich to show that the APJ's decision should be modified. 37 CFR §1.655(a). After careful review, for the following reasons, we are satisfied that Canich has met that burden and, accordingly, we modify the APJ's decision and GRANT Canich Motion to remove Canich U.S. Patent 5,026,798 and Canich Reissue Application 07/973,107, filed November 6, 1992, from this interference proceeding. As a result, this interference is redeclared to involve only Canich Application 07/676,690 and Stevens' Application 07/545,403. A notice to that effect is attached hereto (see Redclaration, paper no. 682).

First, we note that the parties have agreed that Canich Motion 1 is pending without opposition (see paper no. 562).

Second, Canich has met its burden of showing that claims 1-13 of the Canich '798 patent and claims 1-13 of the Canich reissue corresponding to the count are limited to a process of polymerizing α-olefins into crystalline poly-α-olefins and that such a process defines a separate patentable invention from the general process defined by claims 7-10, 14/10 and 29-32 of Stevens' '403 application corresponding to the count.

Canich moved "under 37 CFR § 1.633(b)(2) for a judgment of no interference-in-fact" between Canich's involved '829 patent and Stevens' '403 Application (paper no. 23). In it (pp. 1-12), Canich argues that

Canich believes that Canich's U.S. patent application Serial No. 676,690 ("the '690 application") is the proper application to be in this interference because the '690 application claims the same general process of polymerizing that the '403 application claims. This belief is based on the fact that the '798 patent, and its claims, are a limited species of the general process of polymerizing -- a species directed toward a process of polymerizing α -olefins (defined as an olefin having 3 to 20 carbon atoms, i.e., excluding ethylene) into stereoregular (i.e., crystalline) α -olefin polymers. A process of polymerizing α -olefins into stereoregular polymers is a separate patentable invention over a general process of polymerizing olefins because the crystalline poly- α -olefin requires the catalyst to have stereoregular control over the α -olefin insertion, a feature not found in the general process of polymerizing. It should be noted that the '403 application does not describe, exemplify, teach or suggest any stereoregular poly- α -olefins. Therefore, Canich has filed Motion No. 1 for judgment between the '798 patent and the '403 application.

Canich Motion 1 was denied by the APJ (Decision on Motions, paper no. 237, pp. 7-8) for the following reasons:

In the motion, Canich urges that there is no interference-in-fact between the Canich involved patent and the Stevens application.

The motion is denied for essentially those reasons set forth in the Stevens et al. opposition paper. Canich urges that Stevens et al. do not indicate that their polymer process results in a crystalline product. However recognition of alleged "crystallinity" of the product of the Canich process does not render that process separately patentable from the Stevens et al. process especially when it is clear from the respective specifications that both parties polymerize α -olefins with the same catalyst-cocatalyst combination and there are no limitations in the claims directed to specific process parameters or specific substituents which involve the production of a separately patentable product.

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The APJ granted that part of Canich Motion 2 (paper no. 24) which requested that Canich Application 07/676,690 be added to the interference (thereby mooting Canich Motion 5 (paper no. 27) which sought to declare an additional interference between the Canich '690 application and the Stevens '403 application) and granted Canich contingent Motion 11 (paper no. 46) seeking to add Canich Reissue Application 07/973,107 to the interference, designating claims 1-13 of the reissue as corresponding to the count. As a result of these actions, the interference was redeclared to include the Canich '690 application and Canich's reissue application. For purposes of our review of Canich Motion 1, we will treat Canich's patent and reissue together and interpret Canich's request for review of Canich Motion 1 as being applicable as well to the claims of Canich's reissue application corresponding to the count. There is no dispute that Canich's '690 application should remain in the interference.

In reviewing the merits of Canich Motion 1, we focus our attention on the parties' claims corresponding to the count. 37 CFR § 1.633(b). In that regard, Canich's '798 patent and reissue claims corresponding to the count all require "producing crystalline poly-a-olefins comprising ... recovering a crystalline poly-a-olefin." Stevens' claims are more broad in scope, directed to preparing a polymer. No claim of Canich's patent and reissue is identical to any claim of Stevens' '403 application.

"An interference-in-fact exists when at least one claim of a party that is designated to correspond to a count and at least one claim of an opponent that is

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designated to correspond to the count define the same patentable invention." 37 CFR § 1.633(j).

Resolution of an interference-in-fact issue involves a two-way patentability analysis. The claimed invention of Party A is presumed to be prior art vis-a-vis Part [sic, Party] B and vice versa. The claimed invention of Party A must anticipate or render obvious the claimed invention of Part [sic, Party] B and the claimed invention of Party B must anticipate or render obvious the claimed invention of Party A. When the two-way analysis is applied, then regardless of who ultimately prevails on the issue of priority, the Patent and Trademark Office (PTO) assures itself that it will not issue two patents to the same patentable invention.

Winter v. Fujita, 53 USPQ2d 1234, 1243 (Bd. Pat. App. & Int. 1999). See also 37 CFR § 1.601(n).

Canich has the burden of proving that it is entitled to a judgment of no interference-in-fact. 37 CFR § 1.637(a). See also Bruning v. Hirose, 161 F.3d 681, 684, 48 USPQ2d 1934, 1937 (Fed. Cir. 1998) [the appropriate standard of proof is the preponderance of the evidence standard]. Accordingly, in order to establish that there is no interference-in-fact, Canich must demonstrate that all the Canich patent and reissue claims corresponding to the count are drawn to a separate patentable invention from every claim of Stevens' application which corresponds to the count. See Heymes v. Takaya, 6 USPQ2d 1448, 1451 (Bd. Pat. App & Int. 1988). Canich, as the movant, has the burden of establishing its contention that there is no interference-in-fact with sufficient evidence. Ibid. Declaring an interference creates a presumption that an interference-in-fact exists. Case v. CPC Int'l Inc., 730 F.2d 745, 750, 221 USPQ 196,

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200 (Fed. Cir.), cert. denied, 469 U.S. 872, 224 USPQ 736 (1984). Accordingly, Canich has the burden of proving otherwise.

Canich's species process of polymerizing α -olefins is not anticipated by Stevens' general process of polymerizing (35 U.S.C. § 102). Accordingly, the question is whether such a process would have been non-obvious (35 U.S.C. § 103) with respect to Stevens' generic method. § 1.601(n). In that regard, Canich explains that crystalline poly- α -olefins are unique among polyolefins in general in that their crystallinity depends on the stereoregularity of the pendant groups on the polymer chain (CB 61-63).

The goal, therefore, is to intentionally polymerize an α -olefin into a poly- α -olefin having a particular stereochemical configuration so that it can meet the desired end use. The '798 patent discloses a process for making crystalline poly- α -olefins with particular catalysts. Those catalysts create a specific electronic and steric environment to influence the orientation of the monomer approach to the active metal site so that either isotactic or syndiotactic polymers are the result. The function of the catalyst to control monomer approach and orientation during polymerization is generally referred as the catalyst site control mechanism for producing stereoregular α -olefin polymers. Thus, even though the catalyst precursors for the crystalline polymerization process are species within the general class of compounds there is nothing within that general class to teach one of ordinary skill in the art which precursors will make a crystalline poly- α -olefin. CB 63.

Canich goes on to explain that

The process of polymerizing α -olefins into a crystalline poly- α -olefin polymer would not have been obvious to one of ordinary skill in the art from the general process of polymerizing disclosed and claimed in the '403 application because there are a unique set of problems presented in obtaining a polymer that has a stereoregular structure. The '798 patent discloses that the catalyst system must be designed with an active metal site that influences the orientation of the pendant group of the α -olefin as it approaches the polymerization site. CB 68.

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Canich (CB 68) directs our attention to the specification in the '798 patent for examples showing polymerizing α-olefins into poly-α-olefins with catalysts with different types of mono-Cp ligands. Canich points out that Stevens' '403 application also provides examples that produce polymers with some crystallinity but not crystalline poly-α-olefins. According to Canich (CB 70-71), "obtaining a crystalline poly-α-olefin was surprising from the very start ... Claims 1-13 of the '798 patent reflect the surprising results by requiring recovery of a crystalline poly-α-olefin."

We are satisfied that Stevens' '403 application provides no suggestion to employ the necessary Cp-catalyst to produce crystalline poly-α-olefins from α-olefins as described by each of the claims of Canich's '798 patent and reissue application. Given the disclosure of Stevens' '403 application and the state of the art, one of ordinary skill would not have been led to the process claimed by Canich and even if one of skill would have used α-olefins in the Stevens' process, there is nothing to guide that person to the particular catalyst that would yield a crystalline poly-α-olefin. Accordingly, we find that Canich's process claimed in its '798 patent and reissue application is unobvious over each of the claims of Stevens' '403 application corresponding to the count.

For the foregoing reasons, we modify the APJ's decision and GRANT Canich Motion 1. Accordingly, the Judgment will reflect this finding of no interference-in-fact with respect to Canich U.S. Patent 5,026,798, filed on September 13, 1990, and Canich

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Reissue Application 07/973,107, filed November 6, 1992.

PRIORITY

It is not the burden of the Board to scour the record, research any legal theory that comes to mind and serve generally as an advocate for a party. Compare Ernst Haas Studio, Inc. v. Palm Press, Inc., 164 F.3d 110, 112, 49 USPQ2d 1377, 1379 (2d Cir. 1999). Accordingly, in making our determination as to priority we have reviewed only those specific facts, issues, and arguments of the parties relied upon in their briefs. See 37 CFR §§ 1.656(b)(5) and(b)(6).¹⁴

Stevens has been accorded senior party status and Canich has not challenged that status. Therefore, as the junior party, Canich has the burden of proof of establishing priority.

Stevens relies solely on a priority date of August 31, 1989 as its proof of a constructive reduction to practice (SB xxxi). Accordingly, Canich is under a burden of proof to establish a date of invention prior to August 31, 1989. Canich must establish that either it actually reduced to practice the invention of the count before August 31, 1989, or it first

¹⁴ 37 CFR § 1.656(b)(5) requires:
[A] statement of the facts, in numbered paragraphs, relevant to the issues presented for decision with appropriate references to the record.

37 CFR § 1.656(b)(6) requires:
[A]n argument, which may be preceded by a summary, which shall contain the contentions of the party with respect to the issues it is raising for consideration at final hearing, and the reasons therefor, with citations to the cases, statutes, other authorities, and part of the record relied on.

conceived the invention prior to that date and proceeded with reasonable diligence from a time just prior to the opponent entering the field toward a reduction to practice, either actual or constructive. 35 U.S.C. § 102(g). Haskell v. Colebourne, 671 F.2d 1362, 1365, 213 USPQ 192, 194 (CCPA 1982).

Canich¹⁵ seeks only to establish priority based on an actual reduction to practice of the invention of the count prior to August 31, 1989.

In an interference proceeding, a party seeking to establish an actual reduction to practice must satisfy a two-prong test: (1) the party constructed an embodiment or performed a process that met every element of the interference count, and (2) the embodiment or process operated for its intended purpose.

Eaton v. Evans, 204 F.3d 1094, 1097, 53 USPQ2d 1696, 1698 (Fed. Cir. 2000).¹⁶ The first prong of the test requires a showing of both an actual preparation and a recognition of the embodiment of the count and the second prong of the test requires a showing of a recognition of a specific practical utility for that embodiment.¹⁷ Furthermore, an inventor's

¹⁵ Citing a number of decisions, Canich states that it must "establish by a preponderance of the evidence that at least a single species within the count of the invention claimed in the junior party's patent application was reduced to practice prior to the earliest filing date [i.e., August 31, 1989] that the senior party is entitled to the benefit of relative to the interference count" (CB 40).

¹⁶ "[A] party cannot obviate the initial requirement that a constructed embodiment include every element of the count through evidence that the embodiment operated for its intended purpose, regardless of the quality of such evidence. Put simply, these are two distinct requirements and a party must satisfy each one to establish an actual reduction to practice." Eaton v. Evans, 53 USPQ2d 1696, 1699 (Fed. Cir. 2000).

¹⁷ "[R]eduction to practice requires a showing of three elements: (i) production of a composition of matter satisfying the limitations of the count, (ii) recognition of the composition of matter, and (iii) recognition of a specific practical utility for the composition." Estee Lauder v. L'Oreal S.A., 129 F.3d 588, 592, 44 USPQ2d 1610, 1613 (Fed. Cir. 1997), citing Standard Oil Co. (Indiana) v. Montedison, S.P.A., 494 F. Supp. 370, 206 USPQ 676, 689 (D.Del. 1980), aff'd, 664 F.2d 356, 212 USPQ 327 (3rd Cir.

testimony must be corroborated by independent evidence in order to establish an actual reduction to practice. Knorr v. Pearson, 671 F.2d 1368, 1373, 213 USPQ 196, 200 (CCPA 1982), citing Reese v. Hurst, 661 F.2d 1222, 1225, 211 USPQ 936, 940 (CCPA 1981).

"With regard to the first prong, this Court's well-established precedent requires that the ... performed process include the precise elements recited in the count," Eaton v. Evans, 204 F.3d at 1097, 53 USPQ2d at 1698 (Fed. Cir. 2000). Canich urges that it "presented evidence concerning twelve mono-Cp compounds used with an alumoxane (methylalumoxane or 'MAO') in over 130 polymerization experiments, which amount to over 130 reductions to practice before Stevens' earliest filing date of August 31, 1989" (CB 2) and focuses our attention on the actual reduction to practice of a process of polymerizing using compound AZ and MAO (see CB 44-57).¹⁸ According to Canich (CB 44),

The compound that Canich made first was compound AZ, which has the chemical name: dimethylsilyl(tetramethylcyclopentadienyl)t-butylamido zirconium dichloride; and the chemical formula: $\text{Me}_2\text{Si}(\text{Me}_4\text{C}_5)(\text{N-t-butyl})\text{ZrCl}_2$. This compound was mixed with MAO in a process to polymerize ethylene into polyethylene.

Canich provides a side-by-side comparison of the polymerization process involving AZ and MAO with the subject matter of Count 2 (CB 45-47). It is apparent to us, and Stevens does not dispute it, that a process of polymerizing with AZ and MAO is a species within the scope of Count 2 and includes the precise elements recited in the count. Therefore, a

1981), cert. denied, 456 U.S. 915 (1982).

¹⁸ For this reason, we view Canich's alleged reduction to practice of AZ as representative of all the alleged reductions to practice (e.g., of BZ, CZ, EZ, FZ, GZ, HZ, IZ, IZ2, JZ, and KZ - see CB 31).

process involving AZ could be relied upon as an actual reduction to practice of the invention in interference if, with respect to polymerizing with AZ and MAO, Canich can satisfy the two-prong test.

Reduction to practice is a legal question based on underlying factual determinations. Fujikawa v. Wattanasin, 93 F.3d 1559, 1564, 39 USPQ2d 1895, 1899 (Fed. Cir. 1996). Canich's burden with respect to the facts in trying to prove an actual reduction to practice of AZ is one of a preponderance of the evidence. 37 CFR § 1.657(b). Where "the burden is ... one of a preponderance of the evidence, then appellant's allegations of fact need be supported only by a preponderance of the evidence," Paivinen v. Sands, 339 F.2d 217, 222, 144 USPQ 1, 6 (CCPA 1964).

"[A] preponderance of the evidence ... standard ... only requires the fact finder 'to believe that the existence of a fact is more probable than its nonexistence before [he] may find in favor of the party who has the burden to persuade the [judge] of the fact's existence.' [citing In re Winship, 397 U.S. 358, 371-72 (1970)]."

Bosies v. Benedict, 27 F.3d 539, 542, 30 USPQ2d 1862, 1864 (Fed. Cir. 1994).¹⁹

¹⁹ Compare with the definition given in 5 CFR § 1201.56(c)(2) [revised January 1, 2000] for the preponderance of the evidence standard in administrative proceedings under the Merit Protection Systems Board [cited in Jackson v. Veterans Admin., 768 F.2d 1325, 1329 (Fed. Cir. 1985)]:

Preponderance of the evidence. The degree of relevant evidence that a reasonable person, considering the record as a whole, would accept as sufficient to find that a contested fact is more likely to be true than untrue.

See also St. Paul Fire & Marine Insurance Co. v. United States, 6 F.3d 763, 769 (Fed. Cir. 1993) which states that the "preponderance of the evidence" formulation is the general burden assigned in civil cases for factual matters" and defines "preponderance of the evidence in civil actions to mean 'the greater weight of evidence, evidence which is more convincing than the evidence which is offered in opposition to it' [quoting Hale v. Department of Transportation, 772 F.2d 882, 885 (Fed. Cir. 1985)]."

Accordingly, in order for Canich to prevail, it must establish that, more probable than not, prior to August 31, 1989, the inventor or someone on the inventor's behalf (1) constructed a process of polymerizing an olefin, diolefin or acetylenically unsaturated monomer with an alumoxane (i.e., MAO) and AZ, and recovered a polymer, by showing that (a) AZ was actually prepared and the polymerization and recovery process was actually conducted with AZ and MAO, and (b) that the compound that was prepared was recognized as AZ and that the process that used AZ and MAO was recognized as polymerizing an olefin, diolefin or acetylenically unsaturated monomer and recovering a polymer, and (2) recognized a specific practical utility for the process. Canich's testimony in support thereof must be corroborated. We discuss each of these issues in turn: actual preparation, recognition, utility, and corroboration.

In determining whether Canich has met its burden of proving an actual reduction to practice with respect to polymerizing with AZ, we take into account Stevens' arguments. Stevens disputes that Canich actually made even one mono-Cp compound within the count (SB 99), and, even if Canich did, "Canich failed to perform the analytical studies necessary to establish the identity of a new compound of the type at issue here" (SB 76), and failed to show the "successful use of a catalyst system comprising an alumoxane and a Group 4 metal compound of the required formula" (SB 100). And furthermore, according to Stevens, Canich (SB 100) has failed to show corroboration.

Actual Preparation

To establish actual reduction to practice of AZ and the polymerization and recovery process in which it is used, Canich must first prove "that the inventor actually prepared the composition and knew it would work." Mikus v. Wachtel [II], 542 F.2d 1157, 1159, 191 USPQ 571, 573 (CCPA 1976).

There is no dispute that, on four separate occasions, Canich prepared a compound that Canich alleges to be AZ. According to Canich (CB 22-25), preparation of a compound alleged to be AZ, was first begun on August 16, 1988 and completed on August 25-26, 1988. The compound was also prepared a second (completed September 23, 1988; CB 36), third (September 28-29, 1988; CB 37) and fourth (completed October 20, 1988; CB 38) time. Each of these preparations occurred prior to Stevens' August 31, 1989, priority date.

The August 16-26, 1988 preparation of AZ, which we take as representative, is discussed in detail in Canich's Brief at pp. 22-25, including the equipment used (CB 22), the starting materials (CB 23), and the reaction and precipitation conditions (CB 23-25). We reproduce the steps in the process of preparing AZ as they are described in Canich's Brief:

- "On August 16, 1988, Dr. Canich
 - reacted 230 mls. of degassed n-butyl-Li ... (purchased from Aldrich Chemical) with 40 mls of degassed NH₂-t-butyl ... (also purchased from Aldrich) in pentane using Schlenk techniques and
 - obtained LiHN-t-butyl [1] and the by-product butane." CB 23.
- "On August 17, 1988, Dr. Canich
 - purified the resultant compound by removing the solvent via vacuum in the drybox and
 - ran a proton NMR experiment ... [which NMR spectrum] is clean, showing peaks at the appropriate chemical shifts." CB 23.
- "Starting on August 19, 1988 and finishing on August 22, 1988, Dr. Turner and Mr. Matthew
 - reacted C₅Me₄O with LiAlH₄ in diethylether by adding 60.5 grams of C₅Me₄O dropwise to 235 cc of

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- LiAlH₄ in 500 cc of diethylether using a dropping funnel.
- The solution was refluxed for thirty minutes and
 - then hydrolyzed by adding 100 ml of water dropwise through the dropping funnel.
 - The mixture was then cautiously added to a cold, aqueous 10% sulfuric acid solution.
 - The ether layer was separated out, and
 - the mixture was reduced to about 250 ml by evaporation.
 - To this solution, with stirring was added dropwise 1 ml of concentrated sulfuric acid.
 - The mixture was stirred for 3 hours and
 - after additional work-up, Dr. Turner and Mr. Matthew obtained C₅Me₄H₂.
 - The supporting NMR spectrum shows the appropriate peaks.
 - Straightforwardly, Me₄C₅H₂ [2] was reacted with n-butyl-Li to obtain C₅Me₄HLi [3]." CB 23-24.
 - "On August 23, 1988, Dr. Canich
 - reacted 10 grams of C₅Me₄HLi [3] ... [from Dr. Turner] with 10 ml Me₂SiCl₂ (purchased from Aldrich) ... in tetrahydrofuran ("THF") using Schlenk techniques to obtain 15.13 grams of Me₂Si(C₅Me₄H)Cl [4]." CB 24.
 - "On August 24, 1988, Dr. Canich
 - purified the resultant product by removing LiCl and
 - evaporating off the solvent." CB 24.
 - "On the same day, Dr. Canich
 - obtained a proton NMR spectrum on the product [and]
 - recorded that the NMR 'looks good'." CB 24.
 - "On August 24, 1988, still the same day, Dr. Canich
 - reacted 10 grams ... of Me₂Si(C₅Me₄H)Cl [4] (from above) by adding it dropwise to 3.68 grams ... of NHLi-t-butyl [1] (from above) in about 100 ml of THF.
 - Dr. Canich removed the solvent,
 - precipitated out and
 - filtered off LiCl.
 - Dr. Canich added ether and
 - then 0.095 mole of MeLi dropwise to obtain 7.93 grams of [Me₂Si(C₅Me₄)(N-t-butyl)]Li₂ [5]." CB 24.
 - "On August 25, 1988, Dr. Canich
 - isolated the compound
 - and a proton NMR spectrum was taken on the product. ... The NMR had 'some bubbling', but otherwise shows the existence of the appropriate compound." CB 24.
 - "On August 25, 1988, Dr. Canich
 - suspended 1.13 grams ... of [Me₂Si(C₅Me₄)(N-t-butyl)]Li₂ [5] (from above) in about 100 ml of benzene and
 - slowly added 1.0g ... of ZrCl₄ (purchased from Cerac, Inc.).
 - After stirring overnight,
 - Dr. Canich filtered the mixture to dryness
 - and then extracted it with pentane and
 - placed the solution in the refrigerator at -40°C.
 - After filtration, Dr. Canich obtained 0.25g of Me₂Si(C₅Me₄)(N-t-butyl)ZrCl₂ [6] (compound AZ as yellow solid." CB 25.
 - On August 26, 1988, Dr. Canich
 - recrystallized the product a second time (extraction) and obtained yellow crystals.
 - Two proton NMR spectra of the compound were taken.
 - The first was taken before the second recrystallization, and shows presence of the desired

compound and two major peaks that should not be there.

- The second NMR spectrum was taken after second recrystallization ... and shows all the proper peaks." CB 25.
- On the same day that compound [6] was obtained (August 26, 1988), and
 - on the same laboratory notebook page,
 - Dr. Canich dissolved the 'remains from the side of the good flask' in toluene,
 - added 1 ml of methylalumoxane (obtained from a colleague (Dr. Welborn)) and
 - bubbled ethylene through the solution.
- Dr. Canich observed the formation of polyethylene." CB 25.
- Compound [6] was used to prepare 'stock catalyst' solutions, which were used for several laboratory reactor polymerization experiments from September 1, 1988 through September 9, 1988." CB 25.

Canich (CB 25) maintains that the process described above was an actual preparation of the compound AZ. Stevens disagrees. Stevens (SB 64-66) takes the position that the result of this process, as well as the other attempts²⁰, is not AZ but other unintended compounds. Stevens (SB 65-66) argues that the process Canich used did not actually prepare AZ because it contained certain flaws in its execution:

... there are four basic flaws in the manner in which Canich attempted to synthesize the compounds of the count. These flaws apply to each alleged actual reduction to practice. First, Canich added reagents in the incorrect order. Second Canich added reagents in incorrect ratios. Third, Canich's synthesis experiments were heavily contaminated with impurities. And last, Canich failed to employ the correct analytical procedures necessary to establish the identity and purity of the metal complexes she prepared. ... these errors strongly point to the creation of unintended and unrecognized compounds such as bis-Cp metal complexes, rather than compounds corresponding to the count.

The four flaws in Canich's preparation of AZ that Stevens alleges to have occurred are the only flaws Stevens raises for our consideration. Accordingly, but for these four flaws, Stevens would appear to concede that the August 16-26, 1988 process described

²⁰ "As explained by Dr. Marks, there are four basic flaws in the manner in which Canich attempted to synthesize the compounds of the count. These flaws apply to each of the alleged actual reductions to practice," SB 65.

supra could in fact actually prepare AZ. We now analyze each of these flaws to see if the process described supra, more likely than not, prepared AZ rather than "unintended and unrecognized compounds such as bis-Cp metal complexes."

Stevens: Flaw #1 - Canich Added Reagents In The Incorrect Order

Stevens argues that Canich committed two errors involving adding reagents in the incorrect order.

With respect to the first error, our attention is directed to this step²¹ in the August 16-26, 1988, process (see supra):

- On August 24, 1988, still the same day, Dr. Canich
 - reacted 10 grams ... of $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)\text{Cl}$ [4] (from above) by adding it dropwise to 3.68 grams ... of NHLi-t-butyl [1] (from above) in about 100 ml of THF.

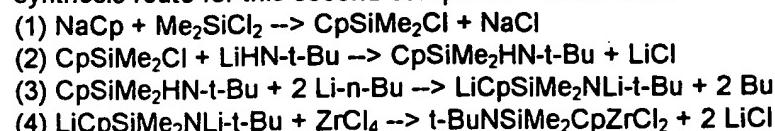
According to Stevens (SB 67):

The first obvious synthesis mistake occurred in step 2.²² As Dr. Marks

²¹ We refer to the particular action taken during the actual August 16-26, 1988 preparation of AZ that corresponds to "step 2" of the synthetic route thought of by Canich (see footnote 22). That particular action occurred on August 24, 1988.

²² Stevens is referring to a synthesis route that Canich has stated it thought of in making AZ.

According to Canich (CB 7-8), Dr. Canich sought to make a zirconium analog of a compound made by Prof. John E. Bercaw (i.e., $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{ScCl}$). Around April 21, 1988, Dr. Canich thought of $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{ZrCl}_2$ (later known as compound AZ) and a way of making it. At the same time, Dr. Canich thought of a different but similar compound ($\text{Me}_2\text{Si}(\text{C}_5\text{H}_4)(\text{N-t-butyl})\text{ZrCl}_2$) and a synthesis route for this second compound - which was:



"The route of synthesis that Dr. Canich thought of for compound AZ was the same, except that the starting compound was not NaCp , but was the lithium salt of tetramethylcyclopentadiene or LiHC_5Me_4 ." CB 8, fact 10.

explained, Canich apparently added the silyl chloride compound dropwise to a solution of the lithium amide compound at room temperature. This procedure was incorrect because it resulted in a local excess of the lithium amide salt. As explained by Dr. Marks, the following unintended compounds are likely to be created under such conditions: [see SB 68-71].

Stevens is arguing that Canich produced a local excess of the lithium amide salt when it dropwise added $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4\text{H})\text{Cl}$ to NHLi-t-butyl .

Canich disagrees. Canich does not dispute that a "local excess of the lithium amide salt" could be a problem in successfully obtaining AZ. However, according to Canich (CRB 114), the "local excess" condition was avoided as a result of "using a slowly dissolving ligand in dilute conditions".

Another technique used by chemists is to carefully select the solvent for the reaction and the amount of solvent relative to the reactant. If a solvent is chosen so that reactant A is fully dissolved in the solvent, but reactant B dissolves into the solvent only at a slow rate, a reaction between A and B will occur as slowly (or only as quickly) as reactant B dissolves into the solvent. ... A variation of this technique was used by Canich in conjunction with the synthesis of Compound AZ. Canich avoided the 'local excess' issue by following a known route for preparing the ligand and by using a slowly dissolving ligand in dilute conditions. CRB 114.

Canich is arguing that there was no "local excess" of the lithium amide salt as a result of the dropwise addition of the $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4\text{H})\text{Cl}$ to NHLi-t-butyl in 100ml of THF.

Accordingly, Stevens and Canich are taking opposite positions on whether a "local excess" of the lithium amide resulted from Canich's dropwise addition of the $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4\text{H})\text{Cl}$. A simple solution for resolving this argument would have been to conduct an experiment whereby $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4\text{H})\text{Cl}$ is dropwise added to NHLi-t-butyl in

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100ml of THF and see if a "local excess" of the lithium amide salt would or would not occur.

However, the panel is not equipped to perform such an experiment. Accordingly, we rely on evidence submitted by the parties. In this regard, it is Stevens who raises the issue.

Therefore, it is incumbent on Stevens to show that a "local excess" of lithium amide salt would more likely than not have been obtained when Canich dropwise added $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4\text{H})\text{Cl}$ to NHLi-t-butyl . That has not been done.

Stevens presents no experimental evidence demonstrating that Dr. Canich, in performing this step in the process, could not, as Canich asserts, have produced $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{Li}_2$. Instead, Stevens (SB 67, footnote 284) directs us to the Declaration of Dr. Tobin J. Marks (SR 162-295, see Paper No. 457) wherein Dr. Marks makes statements to the effect that "it is my opinion that other compounds ... would likely be formed in addition to the intended compound" (see, for example, SR 172, paragraph (43)).

Dr. Marks (SR 172- 173) states that

For step 5 (a) [the step at issue here] ... while the intended compound was $\text{Me}_2\text{Si}(\text{Me}_4\text{C}_5)(\text{N-t-butyl})\text{Li}_2$... it is my opinion that other compounds such as ... would likely be formed in addition to the intended compound. Additionally, compounds such as ... could also be present. Here ... the improper addition of the cyclopentadienyl compound dropwise to the solution of LiHN-t-Bu would have resulted in a large local stoichiometric excess of the lithium salt. Unintended compounds that are likely to form based upon the local stoichiometric excess of LiHN-t-Bu and its reaction with other reagents are shown below [Dr. Marks provides a diagram of the possible unintended compounds resulting from a "local excess"].

Dr. Marks attempts to cast a doubt on Canich's process by raising the possibility that other unintended compounds were produced when Canich performed the step of dropwise

adding $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4\text{H})\text{Cl}$ to NHLi-t-butyl . However, Dr. Marks concludes rather than demonstrates that "the improper addition of the cyclopentadienyl compound dropwise to the solution of LiHN-t-Bu would have resulted in a large local stoichiometric excess of the lithium salt". Dr. Marks does not explain how Canich improperly added the cyclopentadienyl compound and why a "local excess" of the salt would have necessarily formed. There is no evidence that a dropwise addition of $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4\text{H})\text{Cl}$ to NHLi-t-butyl per se produces a "local excess" of lithium salt. Furthermore, it is mere speculation as to whether a "local excess" that might have been formed would result in only unintended compounds. First, there is no evidence that unequivocally shows that unintended compounds were actually made. Second, even if true and, as Dr. Marks submits, Canich obtained unintended compounds, this does not establish that Canich did not also obtain $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{Li}_2$. Both unintended compounds and $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{Li}_2$ could have been obtained and therefore, given that $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{Li}_2$ is an intermediate in AZ's preparation, it still would have been possible for AZ to have been prepared by Canich's August 16-26, 1988, process.

Accordingly, Stevens does not make a persuasive argument that an error was made when Canich performed the step of making $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{Li}_2$.

We reach the same conclusion with respect to the second alleged error raised by Stevens. The second alleged error, according to Stevens, involves the reaction conditions Canich employed in conducting the final step of synthesizing $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{ZrCl}_2$.

(compound AZ). This step of Canich's August 16-26, 1988 process is at issue:

- "On August 25, 1988, Dr. Canich
 - suspended 1.13 grams ... of $[\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})]\text{Li}_2$ [5] (from above) in about 100 ml of benzene and
 - slowly added 1.0g ... of ZrCl_4 (purchased from Cerac, Inc.)."

According to Stevens (SB 71-72),

Canich made a second error in developing the protocol for Canich's step 4.²³ This step is intended to add a single bidentate ligand (the "CpSiMe₂N-t-Bu" in Canich's chart) to each metal to produce the intended bridged metal complex as the dichloride (the "t-BuNSiMe₂CpZrCl₂" in Canich's chart). Once again, however, Canich selected reaction conditions unlikely to make the intended compound and, thus, this provides a second independent basis for the conclusion that Canich failed to prepare the intended metal complexes.

As Dr. Marks explained, appropriate conditions must be chosen in step 4 to avoid surrounding the metal Group 4 tetrachloride with a local stoichiometric excess of the dianion ligand. Otherwise, the likely result is the formation of unintended bis-Cp complexes. The reaction conditions chosen by Dr. Canich in all of the experiments at issue resulted in just such a local excess of the dianion ligand reagent. Because the dianion reagent was partially, or in some instances completely, dissolved in the solution prior to the slow addition of the metal tetrahalide, the most likely product of Dr. Canich's procedures would be bis-Cp complexes even if she used the correct mono-Cp ligand reagent. Indeed, Dr. Canich's selected conditions are exactly those employed when one intends to make bis-Cp complexes.

Stevens is arguing that, during the final step of synthesizing $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{ZrCl}_2$, "the dianion reagent was partially, or in some instances completely, dissolved in the solution prior to the slow addition of the metal tetrahalide" and that this had the effect of producing "a local excess of the dianion ligand reagent". SB 72. Only if the dianion reagent was partially or completely dissolved could there be a local excess of dianion

ligand reagent and only if there is a local excess of dianion ligand reagent could unintended compounds be formed.

While Canich does not dispute that either a "local excess" of dianion ligand might be produced if $[Me_2Si(C_5Me_4)(N-t\text{-butyl})]Li_2$ was partially/completely "dissolved" or that a "local excess" of dianion ligand would have been detrimental to the production of AZ, Canich (CRB 118-119) disputes that $[Me_2Si(C_5Me_4)(N-t\text{-butyl})]Li_2$ was "dissolved". According to Canich, the $[Me_2Si(C_5Me_4)(N-t\text{-butyl})]Li_2$ was "suspended", not "dissolved", thereby precluding both the formation of a "local excess" of dianion ligand and resulting nonintended compounds.

Stevens says that "appropriate conditions must be chosen in step 4 to avoid surrounding the metal Group 4 tetrachloride with a local stoichiometric excess of the dianion ligand" Stevens goes on to say that Dr. Canich chose the wrong conditions. However,, Dr. Canich used the technique of carefully choosing her solvent and its amount so that the dianion ligand was "suspended" in the solvent "Suspended" means not dissolved. Thus, as Dr. Canich added the $ZrCl_4$, the $ZrCl_4$ was able to react only with the dissolved dianion ligand, which was a very small amount. Thus, it was not possible to have a "local excess" of the dianion reagent as Stevens asserts. CRB 118-119.

As with the first alleged error, there is no experimental evidence that would help the panel resolve the issue. Stevens provides no experimental evidence showing that Canich "dissolved" rather than "suspended" $[Me_2Si(C_5Me_4)(N-t\text{-butyl})]Li_2$. We have no concrete evidence showing the formation of a "local excess" under the reaction conditions used by Canich; that is, whenever one "suspends" 1.13 grams of $[Me_2Si(C_5Me_4)(N-t\text{-butyl})]Li_2$ in

²³ See footnote 22.

about 100 ml of benzene.

Instead, Stevens (SB 69, footnotes 302 and 303) directs our attention to the following Canich exhibits (Paper No. 480). We quote the relevant language from each of the exhibits Stevens cites:

- CX 1276 at 41: "suspended in ... Et₂O"
- CX 1373 at 139²⁴: "suspended in ~100 ml benzene"
- CX 1373 at 145: "added to ~ 100 ml toluene"
- CX 1373 at 149: "mixed into ~100 ml ET₂O"
- CX 1373 at 161: "mixed into ~100 ml ET₂O"
- CX 1400 at 3: "suspended in ~ 100 ml Et₂O"
- CX 1400 at 4: "suspended in ~ 100 ml Et₂O"
- CX 1400 at 8: "suspended (largely dissolved) in ~ 100 ml Et₂O"
- CX 1400 at 9: "suspended (largely dissolved) in ~ 100 ml Et₂O"
- CX 1400 at 29: "dissolved suspended in ~ 50 ml Et₂O"
- CX 1400 at 34: "began to dissolve/become suspended in the Et₂O"
- CX 1400 at 35: "suspended in ~ 30 ml Et₂O"
- CX 1400 at 37: "suspended in ~ 100 ml Et₂O"
- CX 1400 at 40: "suspended ... in ~ 100 ml Et₂O"
- CX 1400 at 42: "suspended in ~ 50 ml Et₂O"
- CX 1400 at 43: "suspended in ~ 50 ml Et₂O"
- CX 1400 at 55: "suspended in Et₂O (~ 50 ml)"
- CX 1400 at 56: "suspended in Et₂O (~ 50 ml)"
- CX 1400 at 60: "suspended in ~ 30 ml Et₂O"
- CX 1400 at 61: "suspended in ~ 50 ml Et₂O"
- CX 1400 at 62: "suspended in 40 ml Et₂O"
- CX 1400 at 63: "suspended in Et₂O (~50 ml)"
- CX 1400 at 71: "suspended in ~120 ml Et₂O"
- CX 1400 at 72: "suspended in ~120 ml Et₂O"
- CX 1276 at 39: "diluted in approx 75ml of the solvent, Et₂O"
- CX 1276 at 44: "dissolved in ~ 75 ml of the solvent Et₂O"
- CX 1276 at 45: "dissolved in ~ 75 ml of the solvent Et₂O"

²⁴ The entire sentence reads:

LiMe₄CpSiMe₂N(t-Bu)Li (1.13g = 3.80 4.29 mole) was suspended in ~ 100 ml benzene. 1.0g (4.29 mole) ZrCl₄ was slowly added. The mixture turned a dark yellow upon addition.

Only exhibit CX 1373 mentions benzene. Accordingly, only CX 1373 corresponds to the step in Canich's August 16-26, 1988 process that is at issue here. However, nowhere in the CX 1373 does it state that the ligand was "dissolved". It states, as Canich argues, that the ligand was "suspended". There is nothing in this exhibit to suggest that the "suspended" ligand was in fact "dissolved" in benzene.

The other exhibits (e.g., CX 1400) involve solvents other than benzene and do not suggest that Canich in fact dissolved the ligand. Only CX 1276 at 44-45 unequivocally indicates that Canich "dissolved" the ligand. However, in that preparation, 4 g of ligand were "dissolved" in ~ 75 ml of Et₂O. This compares with the 1.13 grams of ligand that was added to 100 ml of benzene in Canich's August 16-26, 1988, process. It is not at all clear that an analogy can be made to the August 16-26, 1988 process based on the ligand's tendency to dissolve or suspend in the Et₂O. Given that Stevens provides no explanation as to why one would view Et₂O and benzene as equivalent alternatives, comparing these two syntheses, which use different amounts of ligand and solvent as well as different types of solvent, is tantamount to comparing apples and oranges. Accordingly, these exhibits do not provide the necessary support for the argument Stevens has made that Canich "dissolved", rather than "suspended", the ligand when it conducted the August 16-26, 1988 preparation. Therefore, Stevens' argument that Canich produced a "local excess" of dianion ligand is based on mere speculation. Accordingly, Stevens' does not provide a persuasive argument that Canich formed unintended compounds rather than AZ.

Stevens makes two other arguments with respect to this second error.

First, relying on Dr. Marks (SR 179-180), Stevens (SB 72) argues that "appropriate conditions must be chosen in step 4 to avoid surrounding the metal Group 4 tetrachloride with a local stoichiometric excess of the dianion ligand." In that regard, we point out that all that Dr. Marks (SR 179) states is that

Specifically, for step 7(a) of the chart in ¶36 [SR 168], above, while the intended compound was AZ, $\text{Me}_2\text{Si}(\text{Me}_4\text{C}_5)(t\text{-BuN})\text{ZrCl}_2$, ... it is my opinion that many unintended bis(cyclopentadienyl) compounds would, in fact, be the products formed from this reaction procedure. The reason for this is that Canich improperly added the ZrCl_4 to the solution of the alleged dianion reagent which resulted in a large local stoichiometric excess of this reagent. This large local stoichiometric excess of ligand would result in the formation of many unintended bridging ligand complexes.

There is nothing here but opinion and conclusory statements. There is no concrete evidence to persuade us that Canich produced a "large local stoichiometric excess of ligand."

Second, Stevens (SB 73-74) argues that "the formation of bis-Cp zirconium ... metal complexes, following the synthesis procedures used by Dr. Canich, has been fully documented in the literature." One example is, according to Stevens, Du Plooy et al.²⁵ However, there is no indication in this reference of a step of "suspending" $[\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N}-t\text{-butyl})]\text{Li}_2$ in benzene. We therefore cannot conclude, as Stevens has, that this step in Canich's process could produce only unintended bis-Cp complexes and not

²⁵ Karen E. Du Plooy, Ulrich Moll, Sigrid Wocadlo, Werner Massa, and Jun Okuda, "Coordination Properties of Novel Tridentate Cyclopentadienyl Ligands in Titanium and Zirconium Complexes," Organometallics 1995, 14, 3129-3131. See SX 1570.

AZ.

Stevens argues that Canich committed errors involving adding reagents in the incorrect order in two different steps in Canich's preparation of AZ. For the foregoing reasons, we are not persuaded that the evidence shows that Canich more likely than not committed these errors, thereby jeopardizing its ability to prepare AZ when it conducted the steps described supra for its August 16-26, 1988 preparation.

Stevens: Flaw #2 - Canich Added Reagents In Incorrect Ratios

Stevens argues that Canich used starting materials in the wrong ratios. According to Stevens (SB 75-76):

Another problem with Canich's technique results from the fact that the starting compounds used in Canich's syntheses can combine in the intended way, or in unintended ways, depending on the ratios of the starting materials employed. The use of improper ratios results in the formation of impurities in each of the synthetic steps. These impurities can thereafter combine with reagents used in subsequent steps to produce cascades of unintended metal complexes.

An example of Dr. Canich's improper procedure includes the method that she used with lithium alkyls. Dr. Canich explained that lithium alkyl solution was simply added until the solution turned brown. As explained by Dr. Marks, Dr. Canich's technique resulted in the presence of a large excesses of methyl lithium. Because methyl lithium is a known reducing agent, the excess of methyl lithium likely resulted in the reduction of the metal complexes present. This too would have resulted in the formation of unintended metal complexes that are outside the scope of the count. Again, the application of appropriate analytical techniques would have revealed the mistake and changes in procedure could have been implemented.

In arguing that Canich used starting materials in the wrong ratios, it is unclear which

starting materials Stevens is referring to. Stevens refers to Dr. Canich's use of lithium alkyls but it is unclear how this applies to the August 16-26, 1988 preparation that is being analyzed here. Stevens does not explain what the proper ratios for this or any other starting material should have been. Accordingly, it is impossible to determine if this alleged error was committed during the August 16-26, 1988 preparation.

We note Stevens' argument that Dr. Canich used an improper technique when she added a lithium alkyl solution. However, Stevens never explains in what way the technique was improper. Stevens refers to statements made by Dr. Marks (SR 166), for example:

In addition, many of Canich's synthetic experiments specifically indicate that a stoichiometric excess of lithium alkyl reagent was employed. Lithium alkyls are known to reduce the oxidation state of Group 4 metals. Thus, stoichiometrically excess lithium alkyls are likely to result in the formation of greater than expected amounts of paramagnetic species, such as Hf(III), Ti(III) or Zr(III) compounds.

However, Dr. Marks does not actually state that Dr. Canich's technique was improper. Dr. Marks states that a "stoichiometric excess of lithium alkyl reagent" was employed and then speculates that unintended compounds may have been produced. There is no evidence that Dr. Canich's technique was improper per se and that, as a result, Canich's August 16-26, 1988 preparation could not have synthesized AZ but rather could have synthesized only unintended compounds.

Stevens argues that Canich committed an error involving reagents in the wrong ratios. For the foregoing reasons, we are not persuaded that the evidence shows that

Canich more likely than not committed this error, thereby jeopardizing its ability to prepare AZ when it conducted the steps described supra for its August 16-26, 1988 preparation.

Stevens: Flaw #3 - Canich's Synthesis Experiments Were Heavily Contaminated

Stevens argues that Canich's synthesis experiments were contaminated and as a result, there is no guarantee that Canich actually prepared, for example, AZ. According to Stevens (SB 76):

In attempting to synthesize the metal complexes required here, most of the steps must be conducted in an inert atmosphere, such as that provided by an efficient drybox. The evidence showed that Canich's dryboxes were contaminated with oxygen, water and other impurities. Indeed, Dr. Burkhardt, a scientist at the same Exxon research facility and one of Canich's experts here, identified not only oxygen and water, but also carbon monoxide, carbon dioxide, sulfur compounds and acetylene as possible impurities.

The evidence on which Stevens relies in support of its argument is presented in Stevens' Brief at SB 75-82. According to Stevens:

- Most of Canich's synthesis work took place in Dr. Canich's drybox (SB 81 ¶142; relying on CR 7553-68);
- "The mono-Cp metal complexes at issue here are known to be very unstable in the presence of contaminants," (SB 78 ¶138, relying on SR 150-51, SR 8672-75, 8712-13, CR 10272);
- "The evidence shows that Canich's drybox was contaminated" (SB 83 ¶146; relying on SR 248-54); "Indeed, the evidence points to the fact that Canich's dryboxes were highly contaminated," (SB 78-79 ¶138, relying on CR 1789-93; SR 1503-07; CR 1975-76; SR 272-73);
 - "For example, the evidence showed that the inlet and exit ports for the inert gas source were both located on the back wall of the drybox, at the same height, separated by only two feet. This situation creates a 'short circuit' problem in the drybox, a problem that leads to increased contamination from, for example, leaks in the drybox," (SB 82-83 ¶145; relying on CX 597; SX 1561);
 - "Canich noted bubbling in several experiments in which a solution was prepared of a

- sensitive complex, pointing to the reaction of the compound with water and the release of a gas such as methane," (SB 83 ¶146; relying on CR 16-18; CX 1373; SR 172-74);
- "Canich even recorded in her notebook that a compound stored in a sealed vessel in her drybox decomposed visibly within an hour," (SB 83 ¶146; relying on CR 1975-76; CX 1400);
 - "Canich also noted the presence of impurities in her NMR spectra," (SB 83 ¶146; relying on CR 1783-84; 1908-09; 1963-69; 8357-61; 8411-26; 3056; 3103-04; 3124; 3131-32; SX 1568);
 - "Canich also admitted that her drybox was 'heavily full of ether,'" (SB 83 ¶146; relying on CR 1789-93; SR 1503-07);
 - "Canich repeatedly used chlorinated solvents and reagents in her drybox," (SB 84 ¶148; relying on CR 4097-4102);
 - "Volatile compounds can also be introduced ... In recognition of this problem, researchers keep reagents in Sure/Seal™ bottles ... Canich apparently used ordinary bottles for this reagent, rather than Sure/Seal™ bottles, resulting in certain contamination of her drybox atmosphere," (SB 80-81 ¶141; relying on SX 1589);
 - "contamination by laboratory air results in much higher levels of contamination by oxygen than by water. Canich's own expert, Dr. Schrock, identified the possible contamination levels as sufficient to 'rapidly destroy' a 5 mmol sample (about 2 grams) of the metal complex at issue," (SB 79-80 ¶139; relying on CR 4098-12);
 - "Volatile components can enter the drybox ... by diffusion ... leaks ... cracks ... opening the inner door...," (SB 80 ¶140; relying on SR 251-52, CR 4097-98);
 - "Canich ... did not take the appropriate measures to ensure that her drybox atmosphere was free of contaminants that would have destroyed any mono-Cp compound that was present," (SB 78 ¶138, relying on SB 79-86 ¶139-51);
 - "Canich alleges that the purification bed called a "Dri-Train™" would have removed all contaminants from her drybox atmosphere. However ... purification beds [must] be periodically regenerated and replaced. The need to regenerate or replace the purification beds can be determined by monitoring the levels of contamination [especially, according to Stevens, that of oxygen]," (SB 83-84 ¶147);
 - "Canich ... did not monitor the oxygen levels in her drybox. At best, Canich monitored the water levels. ... However, simply monitoring contamination by water is an insufficient check on the amount of oxygen contamination in the drybox atmosphere because, as stated above, oxygen is a more common contaminant ... and the capacity for its removal is lower," (SB 84-85 ¶149, relying on CR 7548-49, 7577-78, 7803-04, 4032, 7595, 7599, 4094-98; SR 3960-62);

Canich responds by arguing that "Dr. Canich and her laboratory assistants followed good laboratory practices when dealing with the air and moisture sensitive compounds at issue here" (CRB 38; see also CRB 121). According to Canich (see, e.g., CRB 41), Stevens' assertions that Canich's dryboxes were contaminated are mere speculation. According to Canich (CRB 45-46),

The Exxon dryboxes were always properly operated, either in a recirculating mode through Dri-trainsTM or in a continuous purge mode using high purity nitrogen. ... almost all of Canich's dryboxes were equipped with moisture meters, some were equipped with oxygen meters, and in every case chemical tests for oxygen and moisture were standard procedure when instrumental methods were not used. ... All Exxon dryboxes that used high purity nitrogen obtained it from the same source ... Therefore, any conceivable oxygen contamination from nitrogen is effectively monitored by a single oxygen meter on the nitrogen supply or in a drybox using nitrogen purge. Any conceivable oxygen contamination from atmospheric intrusion into a drybox would be accompanied by contamination by atmospheric moisture. Therefore, any conceivable oxygen contamination from the ambient atmosphere is effectively monitored by monitoring the moisture level in each drybox. As a result, effective monitoring for water and oxygen contamination in Exxon's dryboxes would require at a minimum the monitoring for oxygen of the nitrogen supply at a single point and the monitoring of moisture in each drybox. In actual fact, the Exxon experimenters went beyond this minimum to monitor for oxygen and moisture in each drybox by instrumental methods or by chemical methods, or by both.

Accordingly, the parties take divergent views with respect to whether Canich, in performing, for instance, the August 16-26, 1988 synthesis, used a contaminated drybox. Both parties appear to agree, however, that a contaminated drybox could hamper the synthesis of AZ.

None of the evidence that Stevens presents establishes that Canich used dryboxes so contaminated that no AZ could possibly be prepared. There is disagreement as to whether any contamination existed within the dryboxes. In this regard, Stevens has not provided any experimental evidence of the levels of contamination that would result from each of the problems that Canich might have encountered. Accordingly, Stevens' assertions that Canich conducted experiments in a contaminated drybox is based on speculation. But even if Stevens were correct and a conclusion of contamination could be made based on, for example, the Canich evidence (SB 83 ¶146) showing that Dr. Canich herself indicated the presence of impurities, the question is not whether there was contamination per se but whether there was contamination sufficient to prevent the formation of compound AZ. That has not been shown.

The question is whether Canich actually prepared AZ. In answering the first prong of the test for determining an actual reduction to practice, Canich must show by a preponderance of the evidence that AZ was actually prepared. Canich cannot show by a preponderance of the evidence that AZ was actually prepared if, as Stevens urges, the drybox was contaminated to a level that prevented AZ from being made. Accordingly, the question of contamination becomes important only if we know, first, the actual amount of contamination in the drybox that Canich used and, second, the maximum allowable level of contamination that would have to be present to prevent AZ from being synthesized. Only by comparing the actual to the maximum allowable levels of contamination can one

determine whether Canich actually prepared AZ in light of any contamination of the drybox. However, there is no evidence of the specific level of contamination that was present in Canich's drybox. There is also no evidence of the level of contamination that would have been necessary to prevent AZ from being formed. Since this information has not been provided, it is impossible to determine whether, when Canich performed its August 16-26, 1988 preparation, Dr. Canich used her drybox in a manner such that Canich was prevented from making AZ.

There is also the matter of whether Dr. Canich's use of a "Dri-TrainTM" removed all contaminants from her drybox atmosphere. Stevens does not dispute that Canich used a "Dri-TrainTM" and does not show that this is either improper or insufficient. Stevens explains that purification beds must be periodically regenerated and replaced to be effective and that monitoring the levels of contamination must still be performed. But Stevens does not demonstrate that Canich did not do so. Stevens is merely pointing out the difficulties surrounding the purification of dryboxes without spelling out in what manner Canich failed to do so. Accordingly, we are not persuaded that whatever atmospheric contamination did exist, Canich did not properly and satisfactorily employ a Dri-TrainTM to remove it.

Stevens argues that Canich's synthesis experiments were contaminated and, as a result, there is no guarantee that Canich actually prepared AZ. For the foregoing reasons, we are not persuaded that the evidence shows that Canich more likely than not contaminated the drybox to such a level that it jeopardized its ability to prepare AZ when it

conducted the steps described supra for its August 16-26, 1988 preparation.

Stevens: Flaw #4 - Canich Failed To Employ Correct Analytical Procedures

The last alleged flaw, according to Stevens (SB 66), is that "Canich failed to employ the correct analytical procedures necessary to establish the identity and purity of the metal complexes she prepared." In particular, Stevens (SB 76-77) argues that

As already discussed at length, Canich failed to perform the analytical studies necessary to establish the identity of a new compound of the type at issue here. In particular, Canich never bothered to conduct routine tests for elemental composition. As a consequence, Canich's analytical data is consistent with the bis-Cp metal complexes that Dr. Marks testified were the likely products of Canich's experiments.

Moreover, as discussed earlier, the quality of Canich's NMR spectra is very poor. Dr. Canich admittedly never bothered to adjust the NMR equipment or to properly reference and maintain the data that were obtained. These problems undermine her ability to rely on the spectra, particularly the observed chemical shifts and integration values to help confirm the identity of the compounds she prepared.

In sum, Canich did not obtain the quality and quantity of analytical data necessary for someone skilled in the art to establish the identity of whatever metal complexes she made. No amount of flawed data (of the type Canich relies upon here) will ever support Canich's asserted synthesis results.

This alleged flaw does not go to the question of whether Canich actually prepared AZ, which is at issue in this section, but to whether Canich recognized or appreciated that the compound it prepared was AZ. Accordingly, we do not address it here. It is more properly addressed in the following section where we review whether Canich has satisfied its burden, by a preponderance of the evidence, to show it recognized or appreciated AZ when it was prepared.

But for the alleged flaws, Stevens does not appear to question the process for actually preparing AZ and then polymerizing an olefin with an alumoxane (i.e., MAO) and AZ and recovered a polymer that Canich describes in its brief. For the foregoing reasons, we are persuaded that the preponderance of the evidence shows that Canich more likely than not actually prepared AZ and used it and MAO to produce and recover a polymer when, for example, it conducted the steps described supra for its August 16-26, 1988 preparation.

Recognition or Appreciation

In order to satisfy the first prong of the test for establishing an actual reduction to practice Canich must also establish that it recognized or appreciated AZ and its use with MAO to produce a polymer when it was prepared. The issue here is whether the preponderance of the evidence shows that Canich confirmed that the product it had obtained was the product they identified as AZ.

Canich asserts that they did enough analytical testing to convince the inventor and others skilled in the art that the inventor in fact prepared the compounds she intended to make. CB 47-48. That testing included performing a proton NMR on August 26, 1988 (CB 25) subsequent to the first synthesis [i.e., the August 16-26, 1988 preparation discussed supra], as well as NMR analyses on other synthesized compounds (CB 29-30), and x-ray crystallographic analyses conducted by Dr. Cynthia S. Day. Day's crystallographic analysis

was conducted subsequent to the fourth synthesis. On March 14, 1989, Dr. Day orally reported the preliminary results of the analysis to Dr. Canich. Final results were reported in August of 1989. Adding to this, Canich relies on NMR spectra for AZ² - a dimethylated version of AZ - obtained from the derivatization work of Dr. Turner, conducted independently of inventor Canich (CB 40), to show that the compound on which it was based had to be AZ. Prior work on similar compounds and subsequent work by others showing inventor Canich's spectra as being consistent with AZ are also relied upon. In conclusion, Canich states that "taken as a whole (including the recently published data, which confirms Canich's data) it is convincing that Dr. Canich in fact made Compound AZ" (CB 50). We agree.

Stevens (SB 12-13) argues that

One of the most glaring deficiencies of Canich's case is Dr. Canich's failure to use adequate analytical techniques to identify reagents, intermediates or products in her experiments. At the top of the list is Canich's failure to perform any recognized and readily available test to determine the elemental composition of the samples through appropriate elemental analysis techniques. These techniques, which include combustion analysis and mass spectrometry, indicate the percentage elemental composition of the compound and, in the case of mass spectrometry, the molecular weight of the compound. These two simple, readily available elemental analysis techniques, in combination with properly performed NMR spectroscopy and x-ray crystallography, would have distinguished between the intended and unintended compounds in every one of Dr. Canich's experiments. Yet, she failed to use these techniques in any of her experiments. In addition, Dr. Canich's demonstrated lack of basic competency in record keeping and careless approach to the use of NMR spectroscopy and other analytical techniques makes Canich's evidentiary record of little or no value. In any event, even if the NMR tests were properly carried out, they could not, without elemental analysis, prove the existence of a metal complex within the

scope of the count. The basic defect applies to all of Canich's alleged twelve reductions to practice and alone defeats Canich's attempt to prove a reduction to practice.

Accordingly, Stevens is presenting two arguments: A) that Canich did not perform an elemental analysis and accordingly could not have known whether the sample it obtained was a compound within the scope of the count, and B) Canich's record keeping is such that there is little or no evidence to substantiate Canich's claim that it in fact made a compound within the scope of the count. Because Canich failed to perform an elemental analysis and failed to properly document the results, according to Stevens, Canich did not recognize or appreciate that it prepared a compound within the scope of the count. We review each of these two arguments in turn.

A) Analytical Testing

Focusing on the August 16-26, 1988 preparation of AZ discussed supra, Canich, in meeting its burden of establishing that it recognized and appreciated that it prepared AZ, relies on two NMR spectra. This is reflected in the following activity of August 26, 1988, reproduced from Canich's Brief:

- On August 26, 1988, Dr. Canich
 - recrystallized the product a second time (extraction) and obtained yellow crystals.
 - Two proton NMR spectrums of the compound were taken.
 - The first was taken before the second recrystallization, and shows presence of the desired compound and two major peaks that should not be there.
 - The second NMR spectrum was taken after second recrystallization ... and shows all the proper peaks." CB 25.

With respect to the NMR spectra, Stevens (SB 12-42) argues that (1) by employing

that technique, Canich was not in accord with standard Exxon procedures and (2) such a technique cannot distinguish between compounds having similar structure.

(1) Elemental Analysis

Stevens (SB 12) asserts that "Canich failed to conduct routine analytical testing necessary to establish what was synthesized." Stevens urges that Canich should have employed elemental analysis since this was a standard technique for identifying new metal complexes.

Stevens directs our attention to the testimony of Dr. Burkhardt, a Senior Staff Chemist at Exxon (SB 14), and Dr. Floyd of Exxon's Materials Characterization Laboratory (SB 14). Dr. Burkhardt (SB 15) testified that a schematic representation of a compound based on NMR spectra may be incorrect. Dr. Burkhardt does not state, as Stevens appears to suggest (SB 15), that NMR spectra are "inadequate" to characterize compounds such as AZ. Depending on other circumstantial evidence, it is quite possible that a schematic representation of a compound based on NMR spectra could be correct. That is apparently the situation here. Canich (CRB 48) is not relying on the NMR spectra as the sole evidence proving that AZ was prepared. For example, Canich (CB 10, 48; CRB 52) points out that Dr. Canich had the benefit of basing the resulting compound as well as its method of preparation on the scandium compounds previously prepared by Dr. Bercaw. This is buttressed by the clear similarity between what Dr. Bercaw prepared - $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-butyl})\text{ScCl}$ - and what Dr. Canich intended to prepare - $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-})$

butyl)ZrCl₂. Regarding Dr. Floyd, the testimony he provides (SB 16-17) establishes that Canich was authorized to make a request to Dr. Floyd for an elemental analysis of the product Canich made. Neither Dr. Burkhardt nor Dr. Floyd have testified that elemental analysis was either necessary or required.

Stevens (SB 17-24) further directs our attention to the testimonies of Dr. Bercaw, an Exxon consultant (SB 17), Dr. Cynthia Day, the president of Crystalytics Company and the person who performed the x-ray crystallographic analyzes mentioned earlier (SB 19-20), Dr. Jordan, a Professor of Chemistry at the University of Iowa, and Dr. Parkin, a Professor of Chemistry at Columbia University. Stevens (SB 25-28) also mentions Dr. Stevens, Dr. Wilson, and other Stevens' experts (i.e., Drs. Marks, Ibers, Landis and Waymouth) and cities publications. In each instance, Stevens seeks to make the case that elemental analysis is the routine, simple, well-established and widely accepted means of identifying a new compound. Stevens also cites Berges v. Gottstein, 618 F.2d 771, 772-73, 205 USPQ 691, 692-93²⁶ (CCPA 1980), one of the facts in the case being that an independent laboratory conducted an elemental analysis to confirm the identity of a compound. According to Stevens, "[e]ven the legal authorities upon which Canich relies endorse the importance of elemental analysis" (SB 28).

There is no dispute that elemental analysis is a routine, simple, well-established and widely accepted means of identifying a new compound. Furthermore, Canich does not

²⁶ "standard elemental analysis determining carbon, hydrogen, and nitrogen percentages."

appear to disagree that elemental analysis was a standard procedure used in Exxon. However, it is not clear why Canich should be limited to that procedure. The question of whether Canich recognized the compound it made was AZ does not turn on whether any specific procedure is the routine, simple, well-established and widely accepted method or the standard used in Exxon. Rather it depends on whether the analytical test used to perform the analysis shows the result to be the compound Canich alleged it to be.²⁷ In our view, that is what Canich has done.

We are not persuaded by Stevens' argument that Canich needed to conduct routine analytical testing necessary to establish what was synthesized. Whether or not Canich recognized or appreciated that it prepared AZ depends not on whether Canich conducted an elemental analysis, as conclusive as such a test might be, but on the evidence as a whole. That evidence, as we have discussed supra, shows more likely than not that Canich recognized and appreciated that it had prepared AZ prior to the August 31, 1989 priority date. Accordingly, Stevens has not persuasively shown that Canich did not recognize or appreciate that it prepared AZ.

(2) NMR

According to Stevens (SB 29), "Canich's proton NMR spectra could not have been used to reasonably identify the new metal complexes Canich alleges were synthesized." In

²⁷ Compare with the standard used in evaluating tests establishing utility: "[T]he inquiry is not what kind of test was conducted, but whether the test conducted showed that the invention would work as intended in its contemplated use." Eastern Rotorcraft Corp. v. United States, 384 F.2d 429, 431, 155 USPQ 729, 730 (Ct. Cl. 1967).

other words, Stevens is arguing that, irrespective of the fact that Canich did not perform an elemental analysis, the NMR spectra provided by Canich does not show the result to be the compound Canich alleges it to be.

Stevens (SB 30-35) discusses a number of problems with respect to Canich's NMR spectra. This includes operator errors (SB 31), that the spectra provide information only about protons (SB 31), the poor quality of the spectrum due to, for example, impurities (SB 30), lack of proper referencing of the spectrum (SB 30-31), improper labeling and maintaining of the spectra (SB 32), potential pulse delay problems (SB 33), poor shimming of the spectra resulting in, for example, poor peak resolution (SB 34), and poor technique as evidenced by different chemical shifts for the same compound tested in the same solvent (SB 35). As a result, according to Stevens, "Canich's proton NMR spectra ... are consistent with the presence of unintended bis-Cp metal complexes that were the likely result of Canich's syntheses" (SB 36). Stevens (SB 36-40) goes on to discuss the similarity one can expect in proton NMR spectra, especially in terms of chemical shifts, for both unintended and the intended compounds.

The evidence does not establish, and Stevens does not dispute it, that Canich's NMR spectra are inconsistent with an identification of AZ. Stevens argues only that Canich's proton NMR spectra are consistent with the preparation of other unintended bis-Cp metal complexes. However, even if that were the case, Canich need not and does not rely solely on the NMR spectra for the analytical testing necessary to establish that it

recognized and appreciated that it prepared AZ.

Consistent with Young v. Bullitt, 233 F.2d 347, 352, 110 USPQ 55, 59²⁸ (CCPA 1956), Canich (CRB 50) submits that the evidence, taken as a whole, establishes "by a preponderance of the evidence that Dr. Canich actually prepared the mono-Cp compounds that she thought she synthesized." Canich (CRB 51) points out that, in addition to the proton NMR, X-ray crystallography was also performed. In this regard, Canich (CB 50-51) argues that Dr. Day's X-ray crystal structure determination of a sample of AZ "is consistent with the X-ray crystal structure data of compound AZ recently published by Dr. Petersen."

Stevens (SB 41-42) responds that (1) the sample Dr. Day characterized is not related to any of Dr. Canich's synthesis experiments and (2) the crystallographic analysis Dr. Day performed could not detect the difference between, for example, a niobium and a zirconium complex. However, Stevens does not dispute that, on February 28, 1989, well before the August 31, 1989 priority date, Dr. Canich sent a letter with a sample alleged to be of AZ to Dr. Day for x-ray crystallographic characterization (CX 310, CR 98-99). Dr. Day began the determination on March 13, 1989 (CB 18) and telephoned Dr. Canich on March 14, 1989, with a final report on August 9, 1989, establishing that the sample had a structure consistent with AZ (CX 188). This too Stevens does not dispute. Given that AZ was prepared four times, the last time in October 1988, and that it was the subject of continuing

²⁸ "[I]t is not necessary that the product relied on by a party ... be identified with absolute certainty, or that a complete analysis should invariably be made. It is sufficient if the evidence, taken as a whole, establishes, with reasonable certainty, the identity of the product."

analyses, through NMR and other techniques, the preponderance of the evidence is that around March 1989 Dr. Day tested a sample that had been earlier synthesized by Dr. Canich. The preponderance of the evidence also supports a conclusion that the sample Dr. Day characterized was a zirconium rather than a niobium complex. The x-ray data Dr. Day provided to Dr. Canich is consistent with later x-ray data provided by Dr. Petersen for the zirconium complex (CB 39). Also, as we noted supra, Canich (CB 48) "was not starting from scratch." Canich sought to prepare a zirconium alternative to the scandium compounds previously prepared by Dr. Bercaw.

Given the evidence taken as a whole, the preponderance of the evidence shows that Canich confirmed through analytical testing, including proton NMR spectra and x-ray crystallographic techniques, that the product it had obtained was the product Canich identified as AZ.

B) Record Keeping

Stevens also argues that Canich's record keeping was such that there is little or no evidence to substantiate Canich's claim that it in fact made a compound within the scope of the count.

The issue of documentation is raised by Stevens (SB 42-64) with respect to:

- laboratory practices as to preparation;
- analytical data (e.g., proton NMR spectra);
- utility (i.e., polymerization);
- corroboration as to preparation; and,
- corroboration as to utility.

At issue here is whether Canich has established with a preponderance of the evidence that it recognized or appreciated AZ when it was prepared. Accordingly, we limit our discussion here to the documentation as to laboratory practices and analytical data and discuss the documentation issue with respect to utility and corroboration infra in the sections entitled "Utility" and "Corroboration", respectively.

According to Stevens, Stevens takes the position that "Canich's synthesis experiments are not properly documented" (SB 51). The reason for this is, according to Stevens, that "Dr. Canich failed to follow Exxon's routine practices and procedures for properly documenting her research efforts" (SB 42). Stevens argues that Canich violated Exxon procedures on how to record analytical data (SB 45). Certain passages referred to as "Exxon's protocol" are reproduced in Stevens' Brief (SB 44-48), wherein Stevens compares Canich's actions to the protocol, concluding therefrom that Canich did not exercise the necessary care to ensure that no question of reliability and authenticity could be raised (SB 47). According to Stevens (SB 48),

Canich's failures to have the experiments properly dated, signed and witnessed has necessitated Canich's attempt to invoke the so-called "cohesive web" theory. However, these same failures, as well as evidence of alteration of data, preclude reasonable reliance on Canich's laboratory notebooks or the analytical results alleged to relate thereto as evidence of priority of invention or discovery.

Canich responds (CRB 24-38) with a discussion of the documentation on record. According to Canich, the documentation on record shows that Dr. Canich's practices

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conformed with Exxon's policy concerning record keeping practices.

The parties disagree as to whether Canich followed standard record keeping practices set forth by Exxon for its researchers. However, the issue here is whether Canich has shown, through contemporaneous records, that it actually prepared, for example, AZ and recognized it before the August 31, 1989 priority date. The issue is not whether Canich violated the rules set forth by Exxon, even though the rules may have been for the purpose of clarifying questions of priority..

In our view, the record is extensive. The record is replete with documents showing in clear and detailed language the steps Canich followed in actually preparing and analytical testing mono-Cp compounds. Based on a preponderance of the evidence, we find that Canich properly documented the results to such an extent that, more likely than not, Canich recognized and appreciated that it prepared a compound within the scope of the count and prepared an olefinic polymer with AZ and MAO.

Utility

Whether a practical utility has been established for a novel compound is a question of fact. To show reduction to practice, the junior party must demonstrate that the invention is "suitable for its intended purpose." Steinberg v. Seitz, 517 F.2d 1359, 1363, 186 USPQ 209, 212 (CCPA 1975).

Canich argues that "each mono-Cp compound was tested for utility as a component

of a polymerization catalyst" (CB 20). According to Canich (CB 20-21), polymerization experiments were conducted and the resulting polymers were tested by gel permeation chromatography. The compounds were active catalysts for olefin polymerization and, according to Canich (CB 21), "typically produced narrow molecular weight distributions." Canich's Brief (CB 26-29) provides a detailed description of a polymerization run with AZ conducted by Mr. Upton on September 1, 1988. Documentation for this run is alleged to be provided by a laboratory notebook and a preprinted sheet taped into the notebook (CB 26). Mr. Upton's run is corroborated by Dr. Canich's "September Update" to Dr. Chow (CX 1339; CR 34-35). Other polymerization runs are also discussed. (e.g., at CB 29-30). This shows that Canich recognized a specific practical utility for an embodiment of the count.

In rebutting Canich's evidence of utility, Stevens argues that "Canich's proofs strongly suggest that bis-Cp metal complexes were responsible for the observed polymerization activity" (SB 106) and that Canich "failed to take any steps to identify ... the components in the catalyst system that were responsible for the polymerization that she reported" (SB 107). Steven's rebuttal is unpersuasive.

There is no dispute that Canich used a compound it had synthesized to conduct the polymerization. Moreover, Stevens provides no evidence that the polymerization activity that occurred when Canich conducted its experiments could not have been obtained from the use of a mono-CP compound. Together with the preponderance of the evidence showing that Canich actually prepared AZ and recognized and appreciated that it had

made AZ, the circumstantial evidence weighs in favor of a conclusion that, when Canich conducted the polymerization, it did so with a mono-Cp compound.

Accordingly, the preponderance of the evidence establishes that the mono-Cp compound Canich prepared operated and was suitable for its intended polymerization purpose.

Corroboration

Canich "must provide independent corroborating evidence in addition to his own statements and documents." Hahn v. Wong, 892 F.2d 1028, 1032-33, 13 USPQ2d 1313, 1317 (Fed. Cir. 1989). Inventor's testimony as to the inventor's activities in reducing an embodiment of the count to practice must be corroborated by some independent evidence. Reese v. Hurst, 661 F.2d 1222, 1226, 211 USPQ2d 936, 940 (CCPA 1981).

Canich (CB 56) argues that it has "provided a cohesive web of corroborative evidence." Canich's case for corroboration is presented in its brief at CB 54-57, supported by facts discussed at CB 5-21. Stevens presents a rebuttal to Canich's position, in part, at SB 108-113. Canich replies to Steven's rebuttal at CRB 67-95.

We turn now to the evidence and determine whether it is sufficient to show that Canich has established a "cohesiveness in the web of allegedly corroborating evidence," Berges v. Gottstein, 618 F.2d 771, 776, 205 USPQ 691, 695²⁹ (CCPA 1980). In doing so,

²⁹ "A different situation existed in Mikus v. Wachtel, *supra*, [Mikus v. Wachtel, 542 F.2d 1157, 191 USPQ 571 (CCPA 1976)] cited by the Board as requiring acts entirely independent of the inventor. At first glance, the problem there appears similar to the instant one. An unwitnessed notebook was

we apply a "rule of reason," Anderson v. Pieper, 442 F.2d 982, 985, 169 USPQ 788, 790 (CCPA 1971). We remain mindful that "[c]orroboration is not a ritual but a method for determining the veracity of the testimony," Mattor v. Coolegem, 530 F.2d 1391, 1395, 189 USPQ 201, 203 (CCPA 1976).

Actual Preparation

According to Canich, corroboration for the actual preparation of a mono-Cp compound within the scope of the count, such as AZ, is established by, for example, the following:

- Dr. Chow (CR 137) declares that Dr. Canich joined his Polymer Research Group at Exxon in September 1987;
- Dr. Chow (CR 137) asked Dr. Canich to prepare a research proposal along the lines of the groups areas of interest - i.e., the "EX-300 project" involving group 4 metal bis-Cp compounds - and he (along with Drs. Burkhardt, Hlatky, Turner and Welborn) received such a proposal (CX 1338 and 75) on or about October 23, 1987;
- Dr. Chow (CR 138-139), among others, decided that Dr. Canich begin work on one of her proposed research projects, to work on group 5 metal compounds, but to spend half her time on the EX-300 catalyst system;
- Dr. Chow (CR 140), among others, received a Monthly Update on or about May 2, 1988, from Dr. Canich with respect to her work on the EX-300 project wherein she

submitted with no firsthand knowledge of a synthesis by co-workers. Independent analyses of a compound within the count, however, were performed. A critical difference in Mikus was the lack of cohesiveness in the web of allegedly corroborative evidence [our emphasis] on which Mikus relied. As this court noted, consistency among the individual components of evidence was not clearly established. For example, some of the analyses were performed on compounds not carrying the proper label. Significantly, the inventor had specifically assigned a suffix to his label which denoted the completion of a necessary process step in the synthesis of the composition of the count. Such a situation does not exist in this case.

Nor can we agree with the board's statements implying that corroborators must have been present at the actual work site or have known something about it other than what was reported to them, for which it cited Patterson v. Clements, 30 CCPA 1262, 136 F.2d 1002, 58 USPQ 539 (1943), a case involving a single corroborating witness. Corroborative testimony does not necessarily have to be an actual witnessing of the reduction to practice by one who understands what is going on in order to be adequate. Sufficient circumstantial evidence of an independent nature can satisfy the corroboration rule."

states "Zirconium chemistry was initiated ... [t]he plan of attack ... will most likely include ... mono-Cp zirconium complexes ... which may or may not be bridged to the Cp ring" (CX 1285);

- Dr. Chow (CR 141) declares that Dr. Canich, also around May 2, 1988, "also reported about a scandium containing compound prepared by Bercaw et al. that reportedly polymerizes propylene";
- a copy of a purchase requisition form (CX 81) prepared by Dr. Canich and approved by Dr. Chow (signed April 21, 1988) shows certain chemicals to be purchased from Aldrich which Dr. Chow (CR 142) declares "were to be used to prepare the zirconium $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-Bu})\text{ZrCl}_2$ ";
- a meeting (summarized in Dr. Chow's May 17, 1988 memorandum - CX 1353) was held on May 6, 1988 and, according to Dr. Chow's Declaration (CR 142), attended by Drs. Canich, Chow, Burkhardt, Hlatky, Pannell, Turner and Welborn and Mr. Finkelstein, in which Dr. Canich's work on mono-Cp Group 4 compounds was discussed;
- on May 19, 1988, Dr. Hlatky (CR 223, CX 1286) signed a patent memorandum "listing Dr. Canich as the inventor of 'Mono-Cp Group 4 complexes with MAO [methylalumoxane]: Olefin Polymerization Catalysts'";
- the chronology of events set forth above is also corroborated by Dr. Turner (CR 544-557);
- Dr. Chow (CR 148) prepared a weekly update on June 6, 1988 and provided a copy to his superior, Dr. Matagna, wherein it states that "Dr. Canich ('Joann' [sic]) 'is working on the synthesis of the amide bridged zirconium metallocene' and '[t]his is the first mono Cp DICOP that we are attempting to synthesize and it will be used in the EX-300 system as well'";
- Dr. Turner (CR 547) declares that, in the summer of 1988, he became aware that "Dr. Canich was lacking the tetra methyl-cyclopentadienyl compound for use in her experiments for synthesizing mono-Cp compounds", whereupon Dr. Turner
 - called Dr. Bercaw for a preparation of the compound, which was sent by Pamela Shapiro (CX 91);
 - prepared, along with Dr. Welborn and T.A. Mathew, the compound from the preparation sent by Shapiro; and,
 - "gave my portion of the yield to Dr. Canich for her use in preparing the mono-Cp compounds" (CR 548-549);
- laboratory notebooks of Dr. Canich were witnessed and signed by Drs. Welborn, Turner or Hlatky (CX 1373, CX 1400) and monthly updates for June, July and August, 1988, were distributed (CX 1332, 1339, 1340), all indicating Dr. Canich's continued work on and final preparation of the mono-Cp compound; and,
- Dr. Turner (CR 550) declares that, by September 30, 1988, he obtained a quantity of the $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-Bu})\text{ZrCl}_2$ finally synthesized by Dr. Canich and gave this to

Mr. Zamora who, at Dr. Turner's request, performed a dimethylation experiment on the sample (CX 1314).

Recognition or Appreciation

According to Canich, corroboration for the recognition or appreciation of the synthesized mono-Cp compound is established by, for example, the following:

- On March 13, 1989, Dr. Day completed an x-ray crystallographic analysis of a sample received from Dr. Canich and Dr. Day reported to Dr. Canich (CX 188) that the sample was $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-Bu})\text{ZrCl}_2$; and,
- Dr. Turner (CR 550-554) declares that, by way of Mr. Zamora's work, he characterized the dimethylated derivative (i.e., $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-Bu})\text{ZrMe}_2$) of Dr. Canich's compound with proton NMR, and with the assistance of Dr. Eckman did the same with carbon-13 NMR, and thereby verified that Dr. Canich supplied the compound $\text{Me}_2\text{Si}(\text{C}_5\text{Me}_4)(\text{N-t-Bu})\text{ZrCl}_2$.

Utility

According to Canich, corroboration for the utility of the synthesized mono-Cp compound is established by, for example, the following:

- Mr. Upton (CR 601-613) declares that he performed separate polymerization experiments "for Dr. Canich using various mono-Cp compounds prepared and supplied to me by Dr. Canich and using MAO as the cocatalyst during the period from December 1988 through May of 1989" (CR 601).

The corroborating evidence discussed supra do not appear to be in dispute. Stevens argues (SB 109-114) instead that

- "Dr. Canich clearly did not follow the practices and procedures required by Exxon to document and confirm that a metal complex of the count was made." SB 109;
- "Canich did not perform the routine tests necessary to establish the identity or whatever was made." SB 111;
- "Canich also did not follow Exxon's routine practices for making, handling and documenting her research efforts." SB 112;

- "... the credibility of Canich's notebooks, proton NMR and carbon NMR spectra is hopelessly compromised." SB 113.
- "It is also telling that Canich fails to offer any analysis of a retained sample of the compounds that Dr. Canich supposedly made, with no explanation as to why no sample of the many that were claimed to have been made has surfaced." SB 114.

Stevens' arguments against Canich's corroborating evidence are unpersuasive.

These arguments go less to the issue of corroboration than to Canich's position with respect to establishing each element of the two-prong test for demonstrating an actual reduction to practice. We have addressed similar arguments in our discussion supra of the two-prong test. Suffice it to say that the issue here is whether Canich has established independent corroborating evidence. Whether or not Dr. Canich followed, for example, certain previously prescribed procedures is irrelevant to the issue of corroboration.

In our view, we do not see that Stevens fairly and reasonably attacks the issue of corroboration. The "rule of reason" involves a reasoned determination as to the credibility of the inventor's story based on an examination, analysis and evaluation of the record as a whole. Berges v. Gottstein, 618 F.2d 771, 776, 205 USPQ 691, 695 (CCPA 1980). To that end, there is a cohesiveness in the web of allegedly corroborating evidence. Canich provides independent evidence through declarations of researchers other than Dr. Canich, and with supporting documents, to show a chronology of events beginning in September of 1987, when Dr. Canich began work with Dr. Chow, through the conception phase during which a research proposal was submitted with a corresponding meeting, purchase of

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ingredients in April 1988, preparation of an intermediate in the summer of 1988, and finally in August 1988, with the preparation of an embodiment within the count. Similarly, independent evidence has been provided showing that, prior to the priority date of August 31, 1989, Dr. Canich recognized and appreciated what she made and established a practical utility for the synthesized compound. Accordingly, we are satisfied that, based on the record as a whole, the preponderance of the evidence weighs in favor of a conclusion that independent corroborative evidence exists for Dr. Canich's testimony in support of Canich's position that an actual reduction to practice occurred prior to the August 31, 1989 priority date. Canich has, in our view, satisfactorily shown the veracity of Dr. Canich's testimony.

For the foregoing reasons, we find that, more probable than not, prior to August 31, 1989, Canich actually prepared AZ and used it in a process with MAO to produce and recover an olefinic polymer, recognized that the compound it prepared was AZ, and recognized a specific practical utility for it. Canich's testimony in support thereof is sufficiently corroborated. Accordingly, Canich has satisfied its burden and prevails as to the issue of priority.

OTHER ISSUES

- I. Stevens includes a section in its brief entitled "**XIV. Stevens' Claims Are Patentable**" (SB 229-241).

Stevens states

In her decision on preliminary motions, Judge Downey deferred decision on Canich's Motion No. 7 under Rule 633(a). Judge Downey deferred Stevens' request to add its first set of amended claims (i.e., claims 67-74, 87-90, 97, 104, 105 and 110) and Stevens' request to cancel Stevens' original claims 7-10, 14, 25, 26, and 29-32. As to Stevens' belated Rule 633(c)(2) motion, Judge Downey deferred decision as to belatedness, Stevens' request to add claims 129-136, Stevens' request to cancel its first set of amended claims and Stevens' renewed request to cancel Stevens' original claims 7-10, 14, 25, 26, and 29-32. (SB 230)

The Board should grant Stevens' Motion No. 25 requesting leave to file its belated Rule 633(c)(2).motion adding claims 129-136 as corresponding to the count. (SB 233)

As we understand it, Stevens is requesting that claims 7-10, 14, 25, 26, and 29-32, all of which except claims 25 and 26³⁰ are designated to correspond to the count, be canceled and replaced with claims 129-136. Stevens makes this request because "in the event the Board grants such a request, all patentability issues related to Stevens' earlier claims are moot" (SB 230). However, that section of Canich's Brief (CB 77-136) that is devoted to the unpatentability of Stevens' claims has been redacted in accordance with the agreement of the parties. Therefore, Stevens provides us with no good reason to grant the request.

Moreover, we see no reason to make an additional determination with respect to canceling

³⁰ Stevens recognizes that "Because Judge Downey separately held that Stevens' claims 25-26 define a separate patentable invention, she redeclared this interference with only Stevens' claims 7-10, 14 and 29-32 designated as corresponding to the count." SB 230.

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7-10, 14, 25, 26, and 29-32, and replacing them with claims 129-136 to be designated as corresponding to the count given that Stevens has not prevailed on the issue of priority and would therefore not be entitled to claims corresponding to Count 2. Accordingly, Stevens' request to cancel claims 7-10, 14, and 29-32 and replace them with claims 129-136 is

DENIED.³¹

Stevens' Brief (239-241) also discusses the patentability of claim 96 and argues that it should not be designated as corresponding to the count. However, claim 96 is not a part of this interference. Accordingly, we make no determination with respect to claim 96.

II. Stevens includes a section in its brief entitled "**XV. Metal Complexes In The +3**

Oxidation State Are Patentably Distinct" (SB 242-278).

In this section, Stevens states:

The preliminary decision granting Stevens' Motion Nos. 4 is no longer challenged by Canich. In the Preliminary Decision, the APJ ruled that metal complexes in the +3 oxidation state are patentably distinct from the count. See Decision on Preliminary Motions, Paper No. 237, at pages 16-18. Pursuant to the parties' agreement, Steven's Motion No. 4 is no longer opposed and Canich's Motion No. 21 has been withdrawn.

Since no action is being requested, we make no further comment.

³¹ We also point out that a party cannot remove involved claims from an interference and thus avoid their cancellation under 35 U.S.C. 135(a) in the event of a final judgment adverse to that party. See Nelson v. Drabek, 212 USPQ 98, 99 (Comm'r Pats. 1979) and Theeuwes v. Bogentoft, 2 USPQ2d 1378, 1379 (Comm'r Pats. 1986).

III. Stevens includes a section in its brief entitled "XVI. Stevens' Claims Directed To Processes Using Non-Alumoxane Cocatalysts Define A Patentably Distinct Invention" (SB 279-292).

In this section of Stevens' Brief, Stevens (SB 282) states that "Stevens' claim 161 defines a patentably distinct invention and should be designated as not corresponding to the count."

Stevens (SB 282, footnote 687) explains that

Stevens' Motion No. 7, Paper No. 17, requested that claims 29-30 be designated as not corresponding; Stevens' Motion No. 15, Paper No. 117, requested that Stevens' claims 82 and 96 be substituted for claims 29-30; and Stevens' Motion Nos. 30, Paper No. 156, and Stevens Motion No. 39 [paper no. 309] requested that claim 161 be substituted for claims 82 and 96.

Accordingly, this request has its origins in Stevens' Motion 7 (paper no. 17).

Stevens Motion 7 was made pursuant to 37 CFR §1.633(c)(4) and seeks to redefine the interfering subject matter by designating Stevens claims 29 and 30 as not corresponding to the count. The motion was deferred to final hearing (see Decision on Motions, paper 237, pp. 19-26) in order to allow Stevens at al.

(1) to correct their specification by motion as to errors contained therein [the motion to be filed no later than the close of their testimony period] (2) provide a comparison of Nickias experiments where the conditions are the same and the only difference is the cocatalyst employed (3) clarify any other inconsistencies in Table V (4) show that the compounds used in the Neithamer and LaPointe declarations were known Lewis acids and ammonium salts useful as catalysts in olefin polymerization (5) that the compounds selected are representative of the class of Lewis acids and ammonium salts and (6) to prepare, if necessary, a direct comparison of the Lewis acids and ammonium salts with aluminoxanes.

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Stevens responded with Motion 38 (paper no. 364) filed pursuant to 37 CFR §1.635 requesting leave to file Motion 39 and, contingent on the granting of Motion 38, Motion 39 (paper no. 365) filed pursuant to 37 CFR §1.635 moving

to enter amendment "I" in its involved application Serial No. 07/545,403 (1) to correct typographical errors therein, and (2) to have Chum, Devore, Savickas, Gunderson, and Bokota declarations, submitted herewith, considered in connection with the party Stevens et al.'s 37 CFR 1.633(c)(4) motion No. 7 to designate the '403 application's claims 29 and 30 as not corresponding to the count.

Attached to the motion is Amendment "I". Amendment "I", which has been filed as paper no. 41 in Stevens application 07/545,403 but which has not yet been entered, amends the application by deleting, on page 62, Examples 67, 74 and 75 from Table V, as well as footnote e). According to Stevens, footnote 2 at p. 4 of Stevens Motion 7 (paper no. 17), these examples contained errors with respect to the indicated cocatalyst and temperature. These are the errors which the APJ allowed Stevens to correct by this subsequent motion.

In answer to the APJ's other concerns, Stevens has submitted the Chum (SR 608), Devore (SR 592), Savickas (SR 619), Gunderson (SR 604), and Bokota (SR 614) declarations. The Devore declaration, for instance, shows a direct comparison of olefin polymerizations using the Group 4 metal complex with an ammonium salt and with MAO, the result being that the ammonium salt catalyst yielded a polymer with a more narrow molecular weight distribution. The Chum declares that the observed more narrow molecular weight distribution was significant and "would inevitably produce a significant, and

because "if the Board disagrees, those patentability arguments [that were set forth in Canich Motion 7³²; i.e., claims 29-30 are unpatentable under 35 U.S.C. §112, first and second paragraphs, §102, §103, and double patenting; see SB 279] will have to be considered" (SB 292). However, that section in Canich's Brief (CB 77-136) which is devoted to the unpatentability of Stevens' claims has been redacted in accordance with an agreement of the parties. Therefore, Stevens provides us with no good reason to grant the request. Moreover, we see no reason to make an additional determination with respect to substituting claims 82, 96, and 129-136, for claims 29 and 30 given that claims 29 and 30 are no longer in the interference. We also emphasize that "there is a long-standing practice of not permitting an applicant in an ordinary application to file a preliminary motion to add claims to its ordinary application for the purpose of having those claims designated as not corresponding to the count." Winter v. Fujita, 53 USPQ2d 1234 (Bd. Pat. App. & Int. 1999), citing L'Esperance v. Nishimoto, 18 USPQ2d 1534, 1537 (Bd. Pat. App. & Int. 1991).

Accordingly, Stevens' request to substitute claims 82, 96, and 129-136 for claims 29 and 30 is DENIED. We reach the same conclusion with respect to the request to review Stevens motion 41 to add claim 161 (SB 280).

IV. Stevens includes a section in its brief entitled "**XVII. The Titanium Subgenus Is A Separately Patentable Invention**" (SB 293-306).

Therein Stevens (SB 230) states that

³² The parties have withdrawn this motion from consideration (paper no. 561).

The record unquestionably shows that the titanium subgenus is separately patentable from zirconium, hafnium, titanium genus, and that Stevens is the inventor of the titanium subgenus. The Board's Decision should clearly reflect these facts in order to resolve all issues between the parties.

Consequently, the Board should reverse the decision denying Stevens' Motion No. 13 [paper no. 116a] to add the titanium subgenus count. ...

As Stevens (SB 229) acknowledges, the titanium subgenus was the subject matter of the count in a separate interference - 103,819 - declared between Canich's U.S. Patent 5,096,867 and Stevens' 07/545,403 application. Accordingly, a determination has already been made that the titanium subgenus is separately patentable from the zirconium, hafnium, titanium genus involved in this interference. Judgment in interference 103,819, as to the subject matter of the count, i.e., the titanium subgenus, was awarded to Stevens and Stevens was entitled to claims in its 07/545,403 application corresponding to the count. Accordingly, the request to review the APJ's decision in the Decision on Motions denying Stevens' Motion No. 13 to add a titanium subgenus to this interference is MOOT.

V. Canich (CB 174-177) provides a section entitled "Canich's Claims."

This section of Canich's brief has been carefully reviewed but it does not appear to contain a request for relief. This section appears to respond to arguments made by Stevens during the interference proceeding with regard to the unpatentability of Canich's claims corresponding to the count. However, Stevens' brief contains no such arguments and therefore Stevens has abandoned that position (see SB 117-228). Matters not raised in a parties' brief are ordinarily regarded as abandoned. Photis v.

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Lunkenheimer, 225 USPQ 948 (Bd. Pat. Int. 1984). Also, we note that Stevens' Motions 1 and 9, which Canich addresses in the brief (CB 176), have been withdrawn from consideration (paper no. 561). Accordingly, because there is no request for relief, no further comment is necessary.

All the issues having been addressed, we enter Judgment. See Paper No. 683.

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The opinion in support of the decision being entered today was not
written for publication and is not binding precedent of the Board.

Paper No. 683

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE BOARD OF PATENT APPEALS
AND INTERFERENCES

JO ANN M. CANICH
Junior Party,

MAILED

SEP 28 2001

v.

PAT. & T.M. OFFICE
BOARD OF PATENT APPEALS
AND INTERFERENCES

JAMES C. STEVENS, FRANCIS J. TIMMERS,
DAVID R. WILSON, GREGORY F. SCHMIDT,
PETER N. NICKIAS, ROBERT K. ROSEN,
GEORGE W. KNIGHT and SHIH-YAW LAI
Senior Party.

RECEIVED

Patent Interference No. 102,954

OCT - 2 2001

JUDGMENT

FISH & NEAVE - PATENT DEPT.
REFERRED TO _____
NOTED BY _____

Before CAROFF, METZ, and LORIN, Administrative Patent Judges.

LORIN, Administrative Patent Judge.

Having decided all the issues properly raised by the parties in their briefs (see the Board's Final Decision (paper no. 681), and in view of the Redeclaration (paper no. 682)), we now enter judgment in this interference pursuant to our authority under 37 CFR § 1.658(a).

Accordingly, on this record, we hold the following:

I. To reflect the finding of no interference-in-fact, Canich is entitled to the claims of U.S. Patent 5,026,798, filed on September 13, 1990, and Canich Reissue Application 07/973,107, filed November 6, 1992.¹

II. As to the subject matter of the count, judgment is entered in favor of junior party, Jo Ann M. Canich and judgment is awarded against senior party James C. Stevens, Francis J. Timmers, David R. Wilson, Gregory F. Schmidt, Peter N. Nickias, Robert K. Rosen, Gregory W. Knight and Shih-Yaw Lai.

Jo Ann M. Canich, the junior party, is entitled to claims 18-33 of their application 07/676,690, all corresponding to Count 2.

¹ Canich is entitled to the claims of its '107 Reissue Application subject to the examiner determining if the reissue application is proper.

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James C. Stevens, Francis J. Timmers, David R. Wilson, Gregory F. Schmidt,
Peter N. Nickias, Robert K. Rosen, Gregory W. Knight and Shih-Yaw Lai, the senior party,
are not entitled to a patent containing claims 7-10, 14/10, 31-32 of their application 07/
545,403, all corresponding to Count 2.

Marc L. Caroff
MARC L. CAROFF
Administrative Patent Judge

Andrew H. Metz
ANDREW H. METZ
Administrative Patent Judge

Hubert C. Lorin
HUBERT C. LORIN
Administrative Patent Judge

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BOARD OF PATENT
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AND
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Interference No. 102,954

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